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IDENTIFICATION OF TOXIC IMPURITIES IN TECHNICAL GRADES OF PESTICIDES DESIGNATED AS SUBSTITUTE CHEMICALS

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Two different approaches were used to identify potentially harmful impurities present in technical grade pesticides. One approach was a theoretical approach that involved a literature search, study the manufacturing processes, identifying the main reactions and the numerous side reactions that may occur, listing the potential impurities that may result, and estimating the likelihood of the occurrence of such impurities at ug/g or lower levels in the final product. The second approach was experimental and involved the use of combined gas chromatography-mass spectrometry (GC-MS) to identify and quantify the major impurities actually present in technical-grade materials.

In addition to the above two tasks the program also included maintaining,and assaying a repository of selected technical-grade pesticides.

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FOREWORD

The many benefits of our modern, developing, industrial society are accompanied by certain hazards. Careful assessment of the relative risk of existing and new man-made environmental hazards is necessary for the establishment of sound regulatory policy. These regulations serve to enhance the quality of our environment in order to promote the public health and welfare and the productive capacity of our Nation's population.

The Health Effects Research Laboratory, Research Triangle Park, conducts a coordinated environmental health research program in toxicology, epidemiology, and clinical studies using human volunteer subjects. These studies address problems in air pollution, non-ionizing radiation, environmental carcinogenesis and the toxicology of pesticides as well as other chemical pollutants. The Laboratory develops and revises air quality criteria documents on pollutants for which national ambient air quality standards exist or are proposed, provides the data for registration of new pesticides or proposed suspension of those already in use, conducts research on hazardous and toxic materials, and is preparing the health basis for non-ionizing radiation standards. Direct support to the regulatory function of the Agency is provided in the form of expert testimony and preparation of affidavits as well as expert advice to the Administrator to assure the adequacy of health care and surveillance of persons having suffered imminent and substantial endangerment of their health.

This study was done to identify impurities in technical grade pesticides. This was accomplished through an evaluation of theoretical products based upon the mode of manufacture and an actual examination of selected materials for the presence of toxic impurities.

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Acting Director
Health Effects Research Laboratory

ABSTRACT

Two different approaches were used to identify potentially harmful impurities present in technical grade pesticides. One approach was a theoretical approach that involved a literature search, study the manufacturing processes, identifying the main reactions and the numerous side reactions that may occur, listing the potential impurities that may result, and estimating the likelihood of the occurrence of such impurities at μg/g or lower levels in the final product. The second approach was an experimental approach that involved the use of combined gas chromatography-mass spectrometry (GC-MS) to identify and quantify the major impurities actually present in technical-grade materials.

In addition to the above two tasks the program also included maintaining and assaying a repository of selected technical-grade pesticides. At the request of the Project Officer the task of assaying product purity by gas chromatography was substituted for the task of identifying and quantifying impurities by combined GC-MS during the second half of the program.

Thirty-four technical-grade pesticides were included in the repository and stored at -20 °C in most cases. Samples were shipped to 15 different EPA or contractor laboratories whenever requested. By having a single lot of each pesticide stored under conditions that would prevent any significant decomposition, each laboratory studying a given pesticide would be supplied identical material and thus permit more meaningful intercorrelation of data generated.

The theoretical evaluations showed that many dozens or even hundreds of specific compounds may be present as impurities in technical-grade pesticides at levels above 1 μg/g. The potential hazard of these possible contaminants can only be speculated upon based on known toxicants of similar chemical structure as a basis. Many of the phosphorus-containing impurities possibly present in organophosphate pesticides may be highly toxic as is known to be the case for paraoxon and tetraethylpyrophosphate. N,N-Diisopropylnitrosamine is a potentially highly carcinogenic compound that is a potential contaminant in trifluralin. Analogs of tetrachlorodibenzodioxin, one of the most toxic mutagenic chemicals known, can theoretically be formed during the preparation of parathion, methyl parathion, PCNB, dicamba, and chlorpyrifos.

Gas chromatographic studies showed that as many as 25 impurities may be detected at levels above 100 μg/g in some technical-grade pesticides. Many of the components were identified by GC-MS in technical-grade chlorpyrifos, dicamba, folpet, malathion, ethyl parathion, parathion, PCNB, phorate, and trifluralin. The identified components were ones that were predicted to be present on the basis of the theoretical studies in nearly every case.

The limited data generated on the program has indicated that extensive additional work is needed to assess the potential hazards of impurities present in technical-grade pesticides. In the interest of more efficient use of research funds for this work we have recommended that the following approaches be included in future efforts:

1. Use high-pressure liquid chromatography (HPLC) and glass capillary column GC-MS in analytical studies.

2. Look for specific toxic components as predicted by theoretical evaluations, e.g. dioxins in chlorpyifos, dicamba, and parathion and diisopropylnitrosamine in trifluralin.

3. Use in vitro bioassay methods in conjunction with HPLC fractionation to assess which impurity fractions are potentially hazardous and should be characterized more extensively. This approach should be applied to samples stored under adverse conditions as well as to fresh technical-grade material.

CONTENTS

Abstract iv
Figures. viii
Tables ix

1. Introduction 1
2. Conclusions and Recommendations. 2
3. Pesticide Repository 5
4. Theoretical Evaluations. 7
5. Analytical Studies 8
    GC Determination of Impurities 8
    GC-MS Determination of Impurities. 8

Appendices

A. Literature References

Aldicarb. 23
Aspon 26
Azinphosmethyl. 27
Azodrin 39
Bromacil. 43
Captan 46
Carbofuran. 55
Chlorpyrifos. 57
Ciodrin 62
Demeton 65
Diazinon. 75
Dicamba 93
Dimethoate. 99
Dinoseb 113
Fensulfothion 120
Fenthion. 121
Folpet 129
Malathion 135
Methomyl. 157
Methyl Parathion. 159
Monuron 175
Parathion 183
Pentachloronitrobenzene 207
Phorate 213
Simazine. 221
Trifluralin 229

CONTENTS (Continued)

Appendices

B. Reactions and Potential Impurities

Aldicarb. 232
Aspon 243
Azinphosmethyl. 249
Azodrin 257
Bromacil. 261
Captan 266
Chlorpyrifos. 274
Ciodrin 280
Demeton 286
Diazinon. 291
Dicamba 300
Dimethoate. 305
Dinoseb 313
Fensulfothion 317
Fenthion. 323
Folpet 333
Malathion 341
Methomyl. 348
Parathion 353
Pentachloronitrobenzene 358
Phorate 361
Simazine. 366
Monuron 371
Trifluralin 378

FIGURES

Number		Page
1	GC-MS of Technical-Grade Parathion	11
2	GC-MS of Technical-Grade Methyl Parathion	12
3	GC-MS of Technical-Grade Malathion	14
4	GC-MS of Technical-Grade Phorate	15
5	GC-MS of Technical-Grade Chlorpyrifos	16
6	GC-MS of Technical-Grade PCNB	17
7	GC-MS of Technical-Grade Folpet	19
8	GC-MS of Technical-Grade Trifluralin	20
9	GC-MS of Technical-Grade Dicamba	21
B-1	Synthesis of Aldicarb	232
B-2	Synthesis of Aspon	243
B-3	Main Reactions in the Preparation of Azinphosmethyl	249
B-4	Synthesis of Azodrin	257
B-5	Synthesis of Bromacil	261
B-6	Main Reactions in the Preparation of Captan	266
B-7	Synthesis of Chlorpyrifos	274
B-8	Alternate Synthesis of 3,5,6-Trichloro-2-Pyridinol	275
B-9	Synthesis of Ciodrin	280
B-10	Synthesis of Demeton	286
B-11	Synthesis of Diazinon	291
B-12	Synthesis of Dicamba	300
B-13	Synthesis of Dimethoate	305
B-14	Alternative Syntheses of Dimethoate	306
B-15	Synthesis of Dinoseb	313
B-16	Synthesis of Fensulfothion	317
B-17	Alternate Synthesis of Fensulfothion	318
B-18	Synthesis of Fenthion	323
B-19	Main Reactions in the Preparation of Folpet	333
B-20	Synthesis of Malathion	341
B-21	Synthesis of Methomyl	348
B-22	Main Reactions in the Preparation of Methyl Parathion and Parathion	353
B-23	Synthesis of PCNB	358
B-24	Synthesis of Phorate	361
B-25	Synthesis of Simazine	366
B-26	Synthesis of Monuron	371
B-27	Alternate Syntheses of Monuron	372
B-28	Synthesis of Trifluralin	378

TABLES

Number		Page
1	Technical-Grade Pesticides Included in the Program.	6
2	Purity of Technical-Grade Pesticides	9
B-1	Reactions Occurring During Aldicarb Production	233
B-2	Possible Contaminants in Aldicarb	235
B-3	Reactions Occurring During Production of Aspon	244
B-4	Possible Contaminants in Aspon	246
B-5	Reactions Occurring During Production of Azinphosmethyl	250
B-6	Possible Contaminanats in Azinphosmethyl	252
B-7	Reactions Occurring During Production and Storage of Azodrin.	258
B-8	Possible Contaminants in Azodrin	259
B-9	Reactions Occurring During Production of Bromacil	262
B-10	Possible Contaminants in Bromacil.	263
B-11	Reactions Occurring During Production of Captan.	267
B-12	Possible Contaminants in Captan.	268
B-13	Reactions Occurring During Production of Chlorpyrifos.	276
B-14	Possible Contaminants in Chlorpyrifos.	278
B-15	Reactions Occurring During Production and Storage of Ciodrin	281
B-16	Possible Contaminants in Ciodrin	283
B-17	Reactions Occurring During Production of Demeton	287
B-18	Possible Contaminants in Demeton	289
B-19	Reactions Occurring During Production and Storage of Diazinon.	292
B-20	Possible Contaminants in Diazinon.	294
B-21	Reactions Occurring During Production of Dicamba	301
B-22	Possible Contaminants in Dicamba	302
B-23	Reactions Occurring During Production and Storage of Dimethoate.	307
B-24	Possible Contaminants in Dimethoate.	309
B-25	Reactions Occurring During Production of Dinoseb	314
B-26	Possible Contaminants in Dinoseb	315
B-27	Reactions Occurring During Production of Fensulfothion	319
B-28	Possible Contaminants in Fensulfothion	320
B-29	Reactions Occurring During Production on Fenthion.	324

TABLES (Continued)

Number Page

B-30 Possible Contaminants in Fenthion. 328
B-31 Reactions in Production and Storage of Folpet. 334
B-32 Possible Contaminants in Folpet. 336
B-33 Reactions Occurring During Production of Malathion 342
B-34 Possible Contaminants in Malathion 343
B-35 Reactions Occurring During Production and Storage of Methomyl. 349
B-36 Possible Contaminants in Methomyl. 351
B-37 Reactions Occurring During Production of Parathion and Methyl Parathion 354
B-38 Possible Contaminants in Parathion and Methyl Parathion 356
B-39 Possible Contaminants in PCNB. 359
B-40 Reactions Occurrring During Production of Phorate. 362
B-41 Possible Contaminants in Phorate 363
B-42 Reactions Occurring During Simazine Production 367
B-43 Possible Contaminants in Simazine. 368
B-44 Reactions Occurring During Production of Monuron 373
B-45 Possible Contaminants in Monuron 374
B-46 Reactions Occurring During Production of Trifluralin 379
B-47 Possible Contaminants in Trifluralin 380

SECTION 1

INTRODUCTION

Various pesticides which have been widely and effectively used for many years (e.g. DDT, heptachlor, and chlordane) have been shown to be very persistent and harmful to man or the environment. For this reason the Environmental Protection Agency has restricted the use of such materials in this country. Users have been forced to select substitute pesticides to do the job that was previously done by the restricted materials. As more information is obtained about the long-range biological effects of various pesticides additional materials will undoubtedly be brought under stricter control or banned for specific uses.

Although the EPA is dedicated to the protection of the environment, it recognizes that farmers and other users need the protection afforded by pesticides. Therefore, when a pesticide is banned for use in controlling a pest with significant economic, ecologic or human health consequences, a substitute must be found to replace it. One of EPA's concerns is that the substitute pesticide authorized may be as hazardous or more hazardous than the material it replaced. Consequently EPA has established a substitute chemicals program involving an in-depth study of a large number of pesticides.

The study reported here involved 35 pesticides included in the EPA substitute chemicals program. The primary objective of the study was the identification of harmful impurities present in certain technical-grade materials.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Any technical-grade chemical is expected to contain numerous by-products or impurities at the one percent (10,000 μg/g) level or lower. Our analytical studies have shown that technical-grade pesticides are no exception in that numerous impurities were detected at the 100 to 10,000 μg/g level. The theoretical studies have shown that there may be dozens or even hundreds of different compounds present as impurities in a given pesticide at levels above 1 μg/g. Some of those compounds, e.g. the dioxins that may be present in trace amounts in dicamba and chlorpyrifos and the diisopropylnitrosamine that may be present in trace amounts in trifluralin, are highly toxic and/or carcinogenic.

Several different experimental approaches can be used to study the potential hazard of a technical-grade chemical. The most widely used approach involves biological studies of the technical-grade material under a variety of laboratory conditions to simulate different types of environmental exposure. This is a very reasonable and realistic approach in that it is based on the assumption that any impurity, no matter how hazardous it may be by itself, will exert the same effect under the laboratory conditions that it will under the field conditions of actual use. Any synergistic or antagonistic effects of the various impurities are automatically built into the experiment whether such effects are known or not.

However, the determination of long-term effects, for example carcinogenic effects, fertility effects, and food chain effects, takes many years and is very costly by the above approach. Consequently such long-term studies are frequently incomplete or simply omitted. A classic example is the case of 2,4,5-T in which the very persistent and highly toxic impurity, tetrachlorodibenzodioxin (TCDD), went undetected for many years.

There are several alternative approaches to detecting such components in a technical-grade chemical that should be considered. One approach is an extensive analytical study to determine all trace components present at levels above 1 ppm. Preparative gas chromatography and liquid chromatography would be used to concentrate the impurities. Analytical separations would be carried out by glass capillary column gas chromatography or high-pressure liquid chromatography. Identification of components would be made by mass spectrometry, n.m.r. spectrometry, and/or infrared spectrometry. After the components were identified their potential hazard would be estimated based on available information or on subsequent biological studies. Obviously a great deal of time and money could be spent on such an approach; however, a few month's effort on a technical-grade chemical could be very informative.

The GC-MS studies used in the current program were within the scope of this approach. Unfortunately this was a minor portion of the total program and was performed only as a superficial screening. There was no preconcentration of the impurities and liquid chromatography was not used at all. For future work using this approach we highly recommend the use of high-pressure liquid chromatography (HPLC) and glass capillary column gas chromatography. The use of HPLC will permit the fractionation of all components present including nonvolatile components and components that may thermally degrade during gas chromatography. The use of glass capillary column gas chromatography will provide much better resolution of components and improve the chances of identifying a component by mass spectrometry.

Another approach involves analytical studies to follow-up on theoretical evaluations. This would involve, for example, separation procedures in conjunction with GC-MS studies to determine the amount of specific dioxins in chlorpyrifos, dicamba, and parathion and the amount of diisopropylnitrosamine in trifluralin. This could be done with a minimal amount of effort. We recommend that it be included in future studies.

A third approach, and the one that we recommend most highly, involves a combination of separation procedures and *in vitro* bioassay. In using this approach various modes of high-pressure liquid chromatography would be used to separate impurities into fractions with little likelihood of decomposition. Each fraction would be assayed using *in vitro* mammalian cell toxicity assays and the Ames *Salmonella* mutagenicity assays. Active fractions would be subfractionated. Only those subfractions which showed significant activity would be analyzed in detail to identify the active component(s). This approach would drastically reduce the amount of analytical effort required for a material yet would permit the detection and identification of most of the potentially hazardous components. It is a much more cost effective approach than the first approach which requires a very extensive analytical study. We highly recommend that this approach be used in future programs.

The assay results showing product purity for materials stored at -20 °C were of dubious value. There is, however, considerable merit in studying the rates of decomposition under ambient conditions or at somewhat elevated temperatures. It might be better to study formulated materials, which are more subject to adverse storage conditions, than full-strength technical-grade material in this respect. By studying degradation at ambient or somewhat elevated temperatures not only would the changes be much more pronounced and thus simplify the analytical problem of detecting real differences but the resulting data would relate to actual storage conditions being used.

Certainly degradation products do form in most cases under normal storage conditions and those products could represent a considerably greater environmental hazard than the freshly prepared product. We therefore recommend that storage stability studies be carried out under ambient and elevated temperatures using for comparison purposes reference samples stored at -75 °C. We further recommend that the storage stability studies be combined with high-pressure liquid chromatography separations and a *in vitro* bioassay program as already described to give a particularly cost effective program.

SECTION 3

PESTICIDE REPOSITORY

Thirty-five different pesticides were included in the overall program. These are listed in Table 1 which also shows which tasks were completed for each material. Samples of technical-grade material for all except aldicarb were acquired and stored. The materials were generously supplied by the manufacturers identified in Table 1 upon request at no cost to the project. The amounts obtained for each material ranged from 2 to 25 kg. Most of the material was repackaged in amber glass screw-cap bottles of three different sizes to hold approximately 10g, 100g, or 500g each. The bottles were thoroughly cleaned and Teflon® liners inserted in the caps prior to use to avoid contamination. The arsenic compounds, two of which were in aqueous solution, are reportedly stable indefinitely at 25 °C and were stored at ambient conditions. All of the other materials were stored in freezers at -20 °C. Each bottle was sealed in a individual polyethylene bag containing indicating Drierite® to prevent hydrolysis. The bags were placed in one-gallon paint cans as a precaution against breakage and spillage.

The main purpose of this repository was to supply material to other EPA contractors. By having a single lot of each pesticide stored under conditions that would prevent any significant decomposition, each EPA contractor studying a given pesticide would be supplied identical material. When a request was received, the appropriate size container in its polyethylene bag could be shipped out immediately since no sample transfer was required.

The shipments were handled in accordance with Hazardous Materials Regulation 49 CFR parts 171-177. All of the materials were classed as Poison B. We conformed to DOT Regulation 173.358, which pertains to materials such as liquid parathion. Parathion is representative of the most hazardous pesticides in the repository. The bottle caps were secured by friction tape and the bottles were packed in vermiculite in fiber drums (28 cm inside diameter, Spec. 21C, DOT Regulation 178.224). This also is in accordance with IOTA Packing Note 628.

Shipment was made by either Federal Express or motor freight. A "Poison" label was placed on each outer shipping carton. For Federal Express shipments, their hazardous materials shipping form was completed. For motor freight shipments a certification of compliance was stamped on the shipping bill.

TABLE 1. TECHNICAL-GRADE PESTICIDES INCLUDED IN THE PROGRAM

No.	Name	Source of Material for Repository	Literature Searched	Theoretical Evaluation Made	Pesticide Study Report Issued	Purity Assayed by GC	Impurities Analyzed by GC	Impurities Analyzed by GC-MS
1	Acephate	Chevron				X		
2	Aldicarb		X	X				
3	Aspon	Stauffer	X	X		X	X	
4	Azinphos-methyl (Guthion)	Chemago	X	X				
5	Azodrin	Shell	X	X		X	X	
6	Bromacil	DuPont	X	X	X	X	X	
7	Cacodyllic Acid	Ansul						
8	Captan	Chevron	X	X	X	X	X	
9	Carbofuran	FMC	X					
10	Chlorpyrifos (Dursban)	Dow	X	X	X	X	X	X
11	Ciodrin	Shell	X	X	X	X	X	
12	Dimeton	Chemagro	X	X				
13	Diazinon	Ciba-Geigy	X	X		X		
14	Dicamba	Velsicol	X	X		X	X	X
15	Dimethoate	Cyanamid	X	X	X	X		
16	Dinoseb	Dow	X	X		X	X	
17	Disulfoton	Chemagro				X		
18	DSMA	Ansul						
19	Fensulfothion (Dasanit)	Chemagro	X	X			X	
20	Fenthion	Chemagro	X	X		X	X	
21	Folpet	Chevron	X	X	X	X	X	X
22	Fonofos (Dyfonate)	Stauffer				X	X	
23	Malathion	Cyanamid	X	X	X	X	X	X
24	Methomyl	DuPont	X	X				
25	Methoxychlor	DuPont				X	X	
26	Methyl Parathion	Monsanto Kerr-McGee Stauffer	X	X	X	X	X	X
27	Monuron	DuPont	X	X				
28	MSMA	Ansul						
29	Parathion	Monsanto	X	X	X	X	X	X
30	PCNB	Olin	X	X	X	X	X	X
31	Phorate (Thimet)	Cyanamid	X	X	X	X	X	X
32	Siduron	DuPont						
33	Simazine	Ciba-Geigy	X	X				
34	Trichlorfon	Chemagro						
35	Trifluralin	Elanco	X	X	X	X	X	X

SECTION 4

THEORETICAL EVALUATIONS

Literature references were collected on methods of synthesis, impurities found, and methods of analysis for most of the 35 pesticides through a search of Chemical Abstracts. The references are given in Appendix A.

On the basis of the literature describing the methods of synthesis and impurities found and on the basis of training and practical experience in organic chemical synthesis, flow sheets showing the synthesis routes of many of the pesticides were prepared. Tabulations of the types of reactions possible and the possible by-products (contaminants) resulting from those reactions were also prepared. The likelihood of a possible by-product being present as an impurity in the final technical-grade product was estimated on the basis of the likelihood of its precursor being consumed by a subsequent reaction, and the likelihood of the impurity being removed during a purification step. The estimates consisted of assigning one of three different concentration ranges: (1) probably present only at levels below 1 µg/g, (2) probably present at levels above 1 µg/g but below 100 µg/g, and (3) probably present at levels above 100 µg/g.

In most cases the list of theoretically possible but very unlikely impurities is incomplete and is limited by practical considerations to include only the most likely impurities. Positional isomers are often grouped together although it is recognized that certain compounds in such a group will be much more likely than others. For the many organophosphorus pesticides, a wide spectrum of monomeric and dimeric pentavalent and trivalent phosphorus compounds containing oxygen and/or sulfur is possible. Although many of these compounds may be formed during the manufacturing process, they may also be formed by hydrolysis, oxidation, and/or disproportionation during storage.

The synthesis schemes, including alternate routes in many cases, tabulations of the types of reactions possible, and tabulations of possible contaminants in many of pesticides studied are given in Appendix B. A summary of which pesticides were included in the theoretical evaluation has been given in Table 1.

SECTION 5

ANALYTICAL STUDIES

GC DETERMINATION OF IMPURITIES

The major impurities present in many of the technical-grade pesticides were studied using gas chromatography alone and in some cases in combination with mass spectrometry. Since the main objective of this task was the detection of as many impurities as possible rather than the determination of gross amounts of the pesticide a very concentrated solution (10 percent) was used and the gas chromatograph was operated at as high a sensitivity as practical. The GC analyses were performed using a Varian Model 1740 gas chromatograph having a flame ionization detector and fitted with a 3-m x 2-mm I.D. glass column packed with 3% OV-17 on 100-120 mesh Gas-Chrom Q. The column temperature was programmed from 60 ° to 300 °C at 8°/min. The injector was maintained at 200 °C and the detector at 310 °C. The nitrogen carrier gas flow was 30 ml/min. In most cases carbon disulfide was used as a solvent to minimize interference from the solvent peak. For those compounds that were insufficiently soluble in carbon disulfide (e.g. captan and folpet) N,N-dimethylformamide (DMF) was used as the solvent. On-column injection of a 2 μl sample was used.

The quantitative values were based on peak areas obtained by computer integration. The response factors for the impurities were assumed to be the same as that of the pesticide in each case. It was further assumed that all of the components present were detected by the GC analysis and the sum of the areas of all of the peaks was equated to 100 percent. Although it is recognized that both of the above assumptions could be in error by a factor of 2, an error of that magnitude would not be great enough to affect the interpretation or usefulness of the data.

The amounts of impurities found in 19 of the technical-grade materials by GC analysis are given in Table 2 . The results show that in most cases there are numerous impurities present at the 100 μg/g level (0.01%) or greater and frequently there are impurities present at levels greater than 1%. This is to be expected of course in any technical-grade chemical. The important point is that the presence of the impurities should be recognized and the potential hazards associated with the impurities should be assessed.

GC-MS DETERMINATION OF IMPURITIES

One approach to assessing the potential hazards associated with the impurities present in technical-grade pesticide involves their identification

TABLE 2. PURITY OF TECHNICAL-GRADE PESTICIDES

Pesticide	Manufacturer	Purity of Technical Sample Stated by Manufacturer	Found by GC Analysis 1975[b]	 1976[c]	Purity of Reference Sample[d], %
Acephate	Chevron	93.5	99.6	93.8	99.3
Aspon	Stauffer	93.0	95.5	92.4	98.0
Azodrin	Shell	58.4	60.1	59.8	99.0
Bromacil	DuPont	95.9	97.0	95.4	99.0
Captan	Chevron	–[a]	93.1	83.2	98.6
Chlorpyrifos	Dow	98.9	92.5	96.9	99.0
Ciodrin	Shell	85.0	87.6	92.7	89.0
Diazinon	Ciba Geigy	88.8	–[e]	88.1	88.8
Dicamba	Velsicol	97.1	–[e]	97.1	100.0
Dimethoate	Cyanamid	95.0	101.5	101.5	99.5
Dinoseb	Dow	97.7	–[e]	106.1	99.0
Disulfoton	Chemagro	–[a]	–[e]	103.2	98.8
Fenthion	Chemagro	96.0	97.9	101.3	98.2
Folpet	Chevron	–[a]	89.4	102.6	99.9
Fonofos	Stauffer	96.9	–[e]	96.5	99.8
Malathion	Cyanamid	–[a]	91.4	93.0	99.3
Methoxychlor	DuPont	98.1	96.2	97.9	99.0
Methyl parathion	Monsanto	80.0	74.4	74.6	99.0
Methyl parathion	Kerr-McGee	80.0	74.9	73.8	99.0
Methyl parathion	Stauffer	80.0	78.3	74.1	99.0
Parathion	Monsanto	98.5	97.7	98.0	99.0
PCNB	Olin	99.0	99.0	99.0	99.0
Phorate	Cyanamid	85.0	93.6	90.2	99.9
Trifluralin	Elanco	97.7	99.8	97.2	99.8

a. Not available.
b. Conducted during the period of April 1 to October 31, 1975.
c. Conducted during May and June, 1976.
d. Supplied by EPA.
e. Not analyzed during this period.

by combined gas chromatography-mass spectrometry. This method was used to study seven of the pesticides.

The gas chromatographic conditions used for the GC-MS studies were similar to those described for the GC studies. Finnigan Models 1015 and 3200 quadrupole mass spectrometers equipped with electron impact and chemical ionization sources, respectively, and coupled to Finnigan Model 9500 gas chromatographs were used for this work. The mass spectral conditions as well as the data output were controlled by a Systems Industries Model 150 interactive computer system. Methane was used as the carrier gas for the chemical ionization (CI), which was used to obtain molecular weight data and a minimum of fragmentation. Helium was used as the carrier gas for the electron impact (EI) to give fragmentation patterns to support the tentative identification resulting from the CI work.

Reconstructed gas chromatograms were obtained based on the total ion intensity of each individual mass spectrum. Reference EI mass spectra were obtained from the literature whenever possible. Computerized spectral matching using the original Aldermaston data tape (1972) of 9500 reference EI mass spectra was also applied. The identifications made were based on a combination of molecular weight data, fragmentation patterns, reference mass spectra, gas chromatographic retention times, and a knowledge of expected impurities predicted by the theoretical evaluations.

Parathion

Ten of the 19 impurities detected in technical-grade parathion by GC analysis were tentatively identified by mass spectrometry. The identities are indicated on the reconstructed gas chromatogram, Figure 1, along with the amounts present. All of the ten are components predicted to be present from the theoretical evaluations.

The phosphoryl chloride was an intermediate which we had expected to be more completely hydrolyzed. The three ethyl phosphates were expected from the reaction of P_2S_5 with ethanol. The p-nitrophenol was an intermediate that was expected. The p-nitrophenetole was formed by the reaction of chloronitrobenzene with ethanol. The two minor parathion isomers are very likely a thiolo compound and an ortho-nitro compound. Reference materials corresponding to the impurities or model compounds were not available to help in determining the isomeric identities. The dithio analog of parathion found was also an expected impurity.

Methyl Parathion

The results from the GC-MS study of methyl parathion were similar to those found for parathion. The manufacturing process for methyl parathion is essentially the same as that for parathion except that methyl alcohol is used instead of ethyl alcohol. Seven of the impurities analogous to those found in parathion were identified. These are indicated on the reconstructed gas chromatogram given in Figure 2.

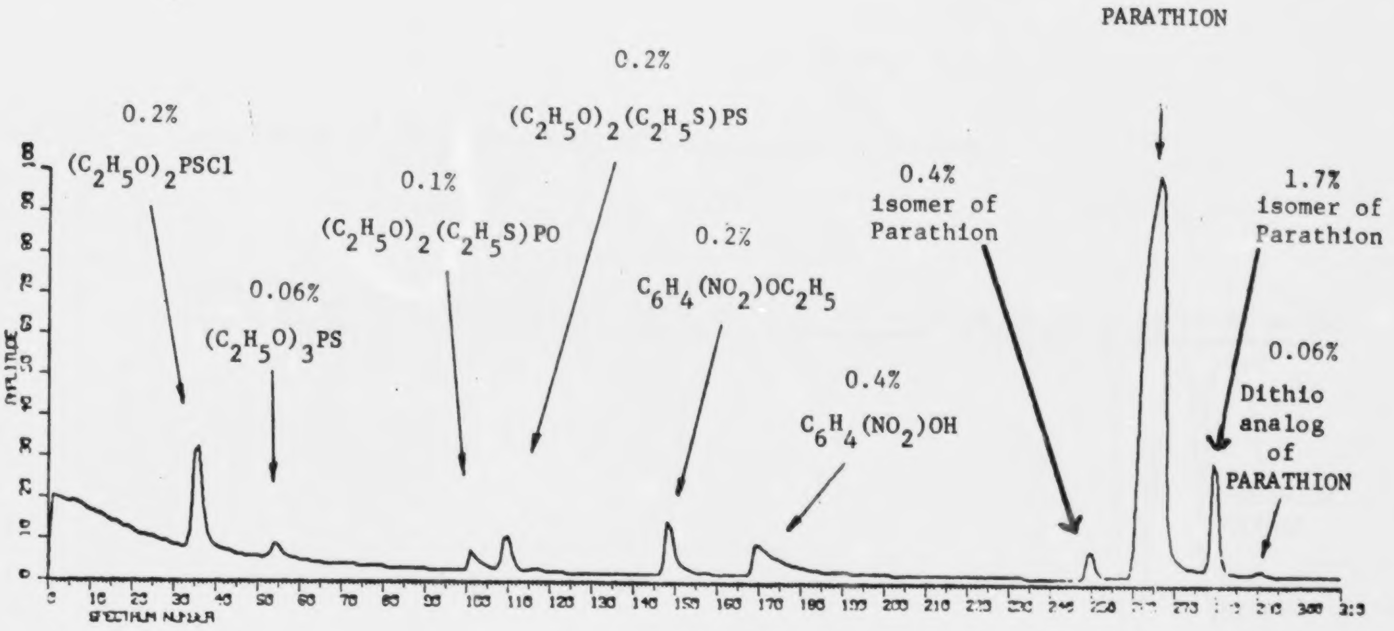

FIGURE 1. GC-MS OF TECHNICAL-GRADE PARATHION

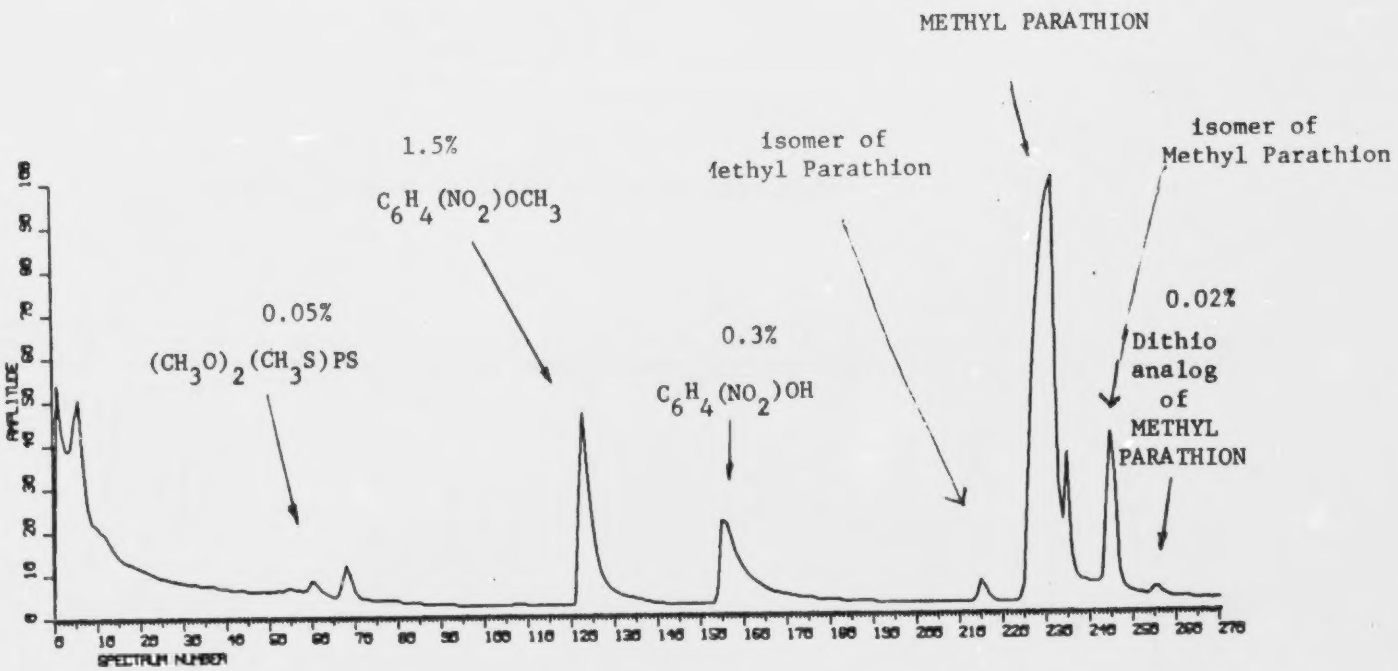

FIGURE 2, GC-MS OF TECHNICAL-GRADE METHYL PARATHION

Malathion

Malathion is another common organophosphate insecticide. In addition to the various possible phosphorus-containing impurities derived from the reaction of methanol with P_2S_5 there is the very likely possibility of impurities derived from maleate intermediates and hydrogen sulfide or methyl mercaptan adducts.

Eight components were identified, including two methyl phosphates, a pyrophosphate, diethyl maleate, and the hydrogen sulfide and methyl mercaptan adducts of diethyl maleate. The minor malathion isomer found is probably the thiolo compound. The identified components and the amounts found are indicated on the reconstructed gas chromatogram given in Figure 3.

Phorate

Phorate, also known as Thimet®, is an organophosphate insecticide which requires formaldehyde and ethyl mercaptan in the manufacturing process. In addition to the formation of the usual phosphate and pyrophosphate by-products, it is quite likely that the hydroxymethyl intermediate will react with a second molecule of the dithioate to give a bis-phosphoryl compound or react with the ethyl mercaptan with loss of water instead of loss of hydrogen sulfide to form the ethoxy analog. Seven different phorate impurities were identified by GC-MS analysis. These were four ethyl phosphates, a pyrophosphate, the ethoxy analog and the bis-phosphoryl compound. These components and the amounts found are indicated on the reconstructed gas chromatogram given in Figure 4.

Chlorpyrifos

Chlorpyrifos, also known as Dursban®, is an organophosphate insecticide analogous to parathion but prepared from 3,5,6-trichloro-2-pyridinol instead of p-nitrophenol. In addition to the usual phosphate and pyrophosphate by-products possible, there is the possibility of forming substituted pyridines and dipyridodioxins during manufacture. The impurities actually identified by GC-MS included two ethyl phosphates, a pyrophosphate, the intermediate trichloropyridinol, an isomer of chlorpyrifos (probably the thiolo isomer), and a tetrachloro analog of chloropyrifos. These are shown in Figure 5.

PCNB

Pentachloronitrobenzene (PCNB) is one of the purest pesticides found in this study. It is prepared by the chlorination and nitration of benzene. The primary impurities expected are, therefore, various other chlorinated and nitrated benzenes. Four such compounds were found by GC-MS analysis as shown in Figure 6. Although trace amounts of sulfonic acids, biphenyls, biphenylenes, dibenzodioxins, dibenzofurans, and diphenyl ethers are theoretically possible as by-products, none was found.

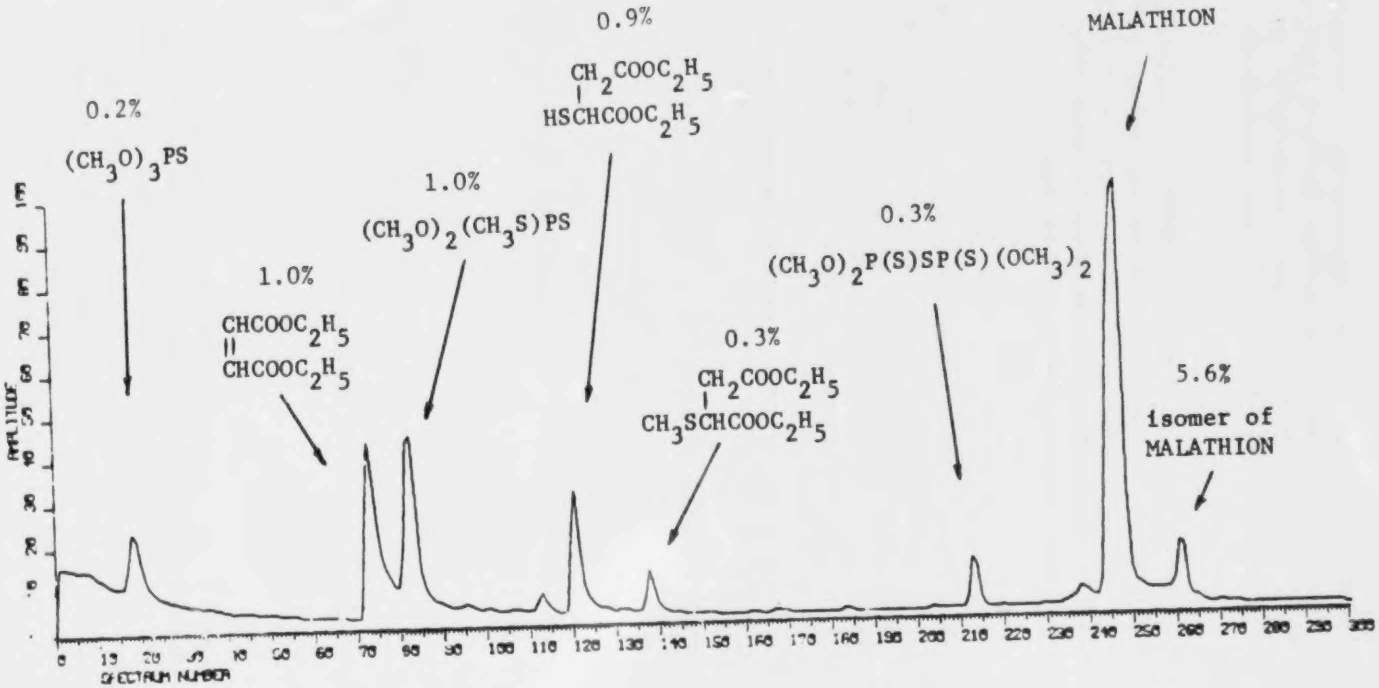

FIGURE 3. GC-MS OF TECHNICAL-GRADE MALATHION

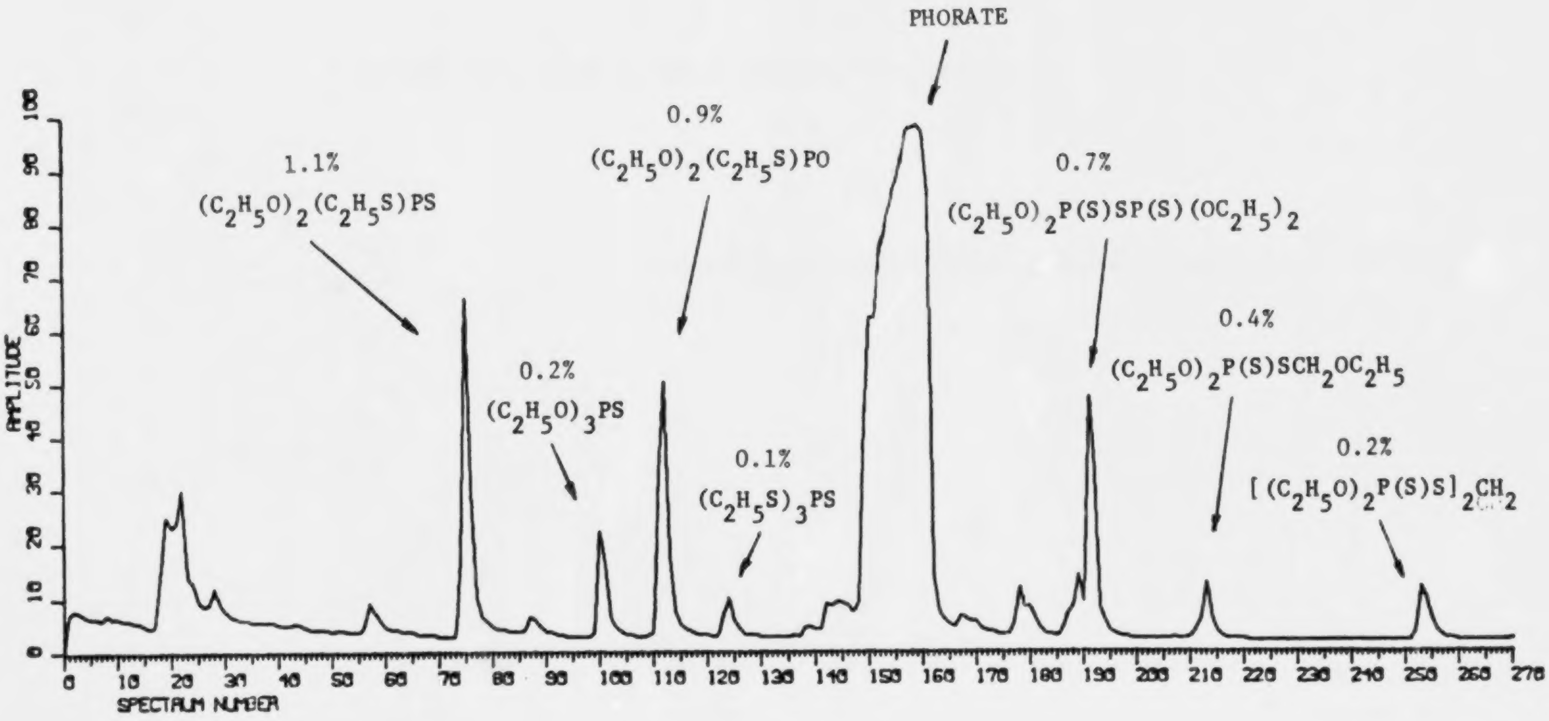

FIGURE 4. GC-MS OF TECHNICAL-GRADE PHORATE

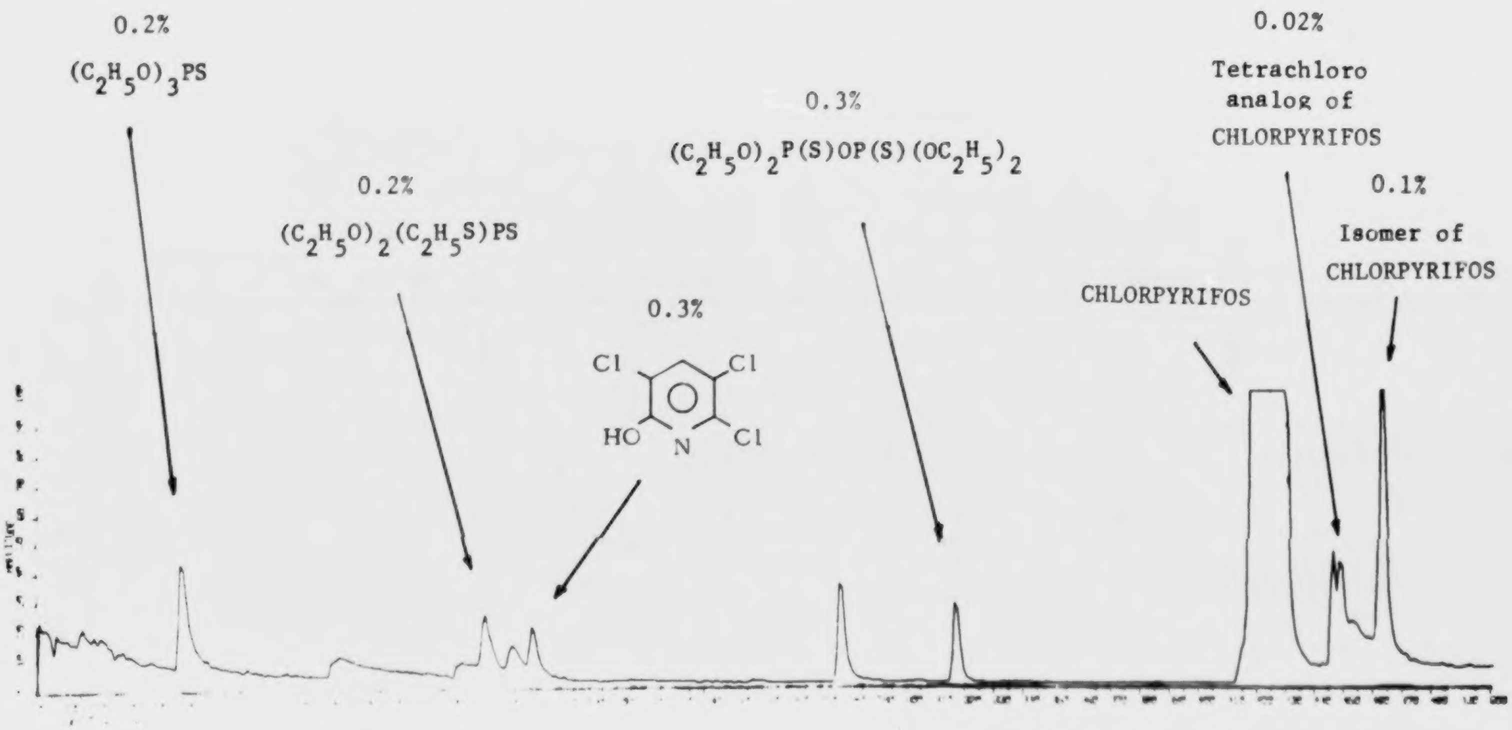

FIGURE 5. GC-MS OF TECHNICAL-GRADE CHLORPYRIFOS

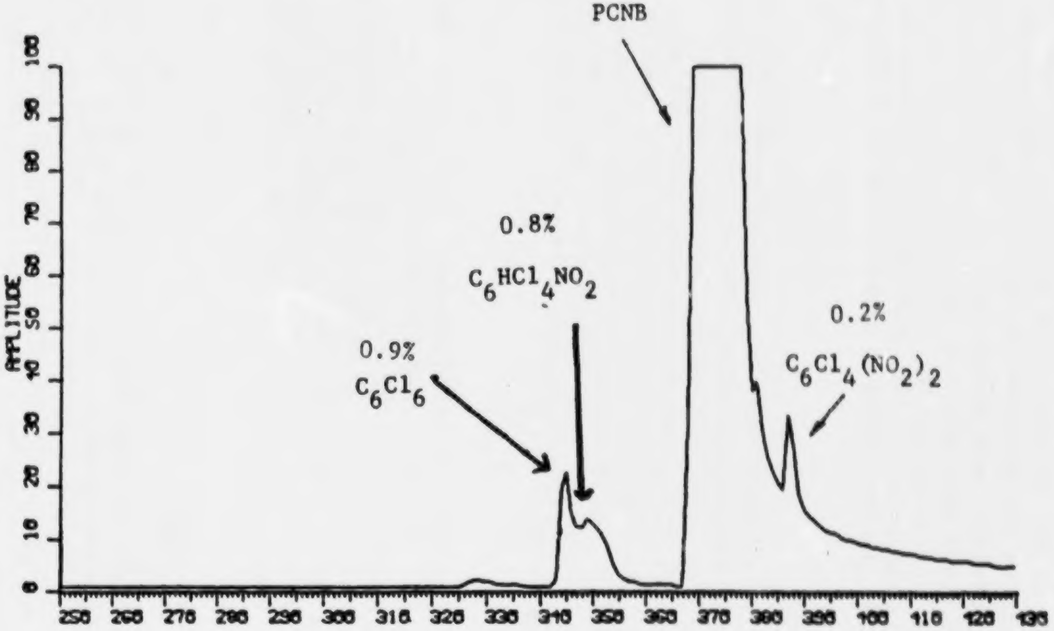

FIGURE 6. GC-MS OF TECHNICAL-GRADE PCNB

Folpet

Folpet is a fungicide prepared from phthalimide and perchloromethyl mercaptan. In addition to derivatives of phthalic anhydride, derivatives of maleic anhydride and oxalic acid can be formed during the oxidation step in the initial part of the manufacturing process. Various sulfenyl, sulfinyl, and sulfonyl impurities are possible as well as acids, amides, imides, chloroamides, chloroimides, and maleic adducts. The major impurities found in the GC-MS study were phthalic anhydride and phthalimide. The N-chlorooxamic acid found was one of the least expected by-products. A small amount of elemental sulfur was also found. The reconstructed gas chromatogram showing the contaminants found is shown in Figure 7.

Trifluralin

Trifluralin is a widely used herbicide prepared from 4-chloro-3,5-dinitrobenzotrifluoride and diisopropylamine. During the overall process including the preparation of the benzotrifluoride there are possibilities for the formation of all sorts of benzene derivatives having chloro, fluoro, nitro, methylol, carboxyl, aldehyde, hydroxyl, ether, amino, and/or substituted amino functionalities. Although 14 components were detected by the GC analysis only six components were identified in the GC-MS study. These were two chloro compounds and four trifluralin isomers as indicated in Figure 8.

Dicamba

Dicamba (3,6-dichloro-2-methoxybenzoic acid) is an herbicide prepared by the methylation of the corresponding hydroxybenzoic acid. Various benzene derivatives having hydroxyl, chloro, and/or carboxyl functionalities are possible as by-products as well as chlorinated dibenzodioxins. The impurities identified by the GC-MS studies included the methyl ester of dicamba, the methyl ester of the hydroxy analog, a dimethoxychlorobenzene, and a dichloromethoxybenzene. These are indicated on the reconstructed gas chromatogram in Figure 9.

GC ASSAY OF PRODUCT PURITY

One of the tasks of this program requested by the Project Officer was the assay of the product purity of some of the materials before and after being stored at -20 °C for a period of time. This study would compare the rate of degradation of a technical-grade sample to that of a reference sample when both are stored at -20 °C. An indication of the presence of impurities which catalyze degradation would thus be obtained.

In this work an accuracy of GC quantification of better than ± 5 percent was desired. Only the GC peak of the pesticide itself was of interest. Therefore, a much more dilute solution was used than for the study of impurities. The solutions were prepared by dissolving 10 to 20 mg of material in DMF containing an appropriate hydrocarbon standard to give a solution containing 20 mg/ml. This solution was then diluted with carbon disulfide to give a solution containing 2 mg/ml (0.2%). By using DMF

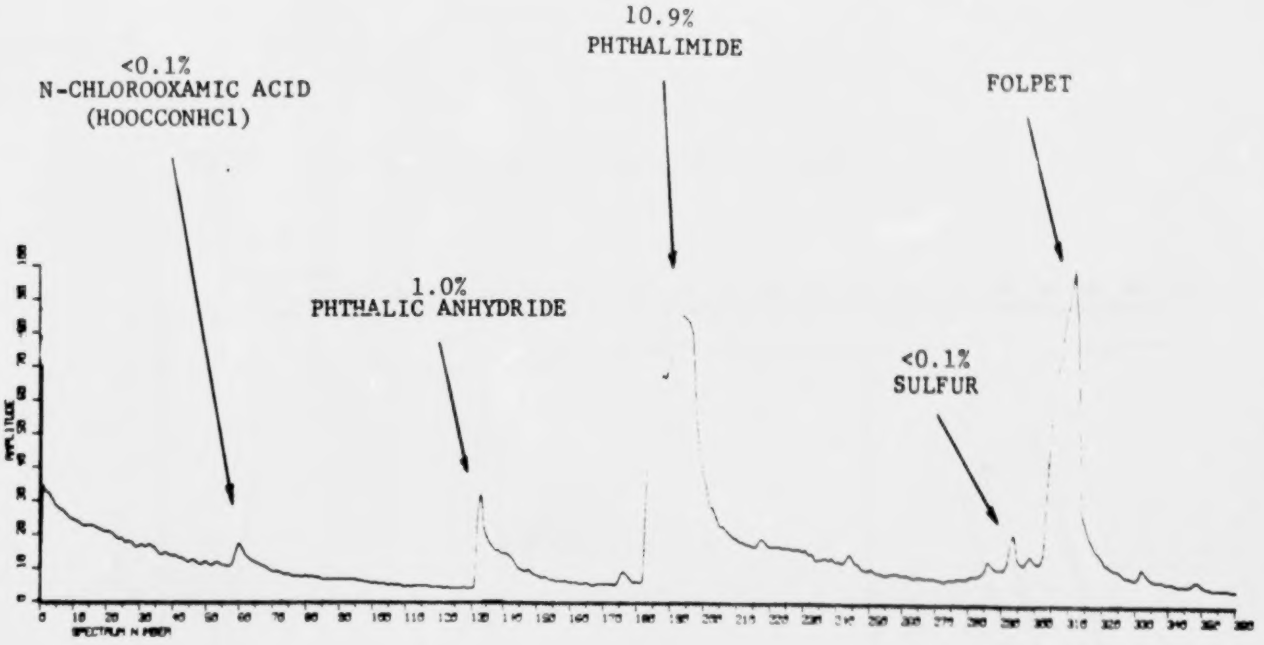

FIGURE 7 . GC-MS OF TECHNICAL-GRADE FOLPET

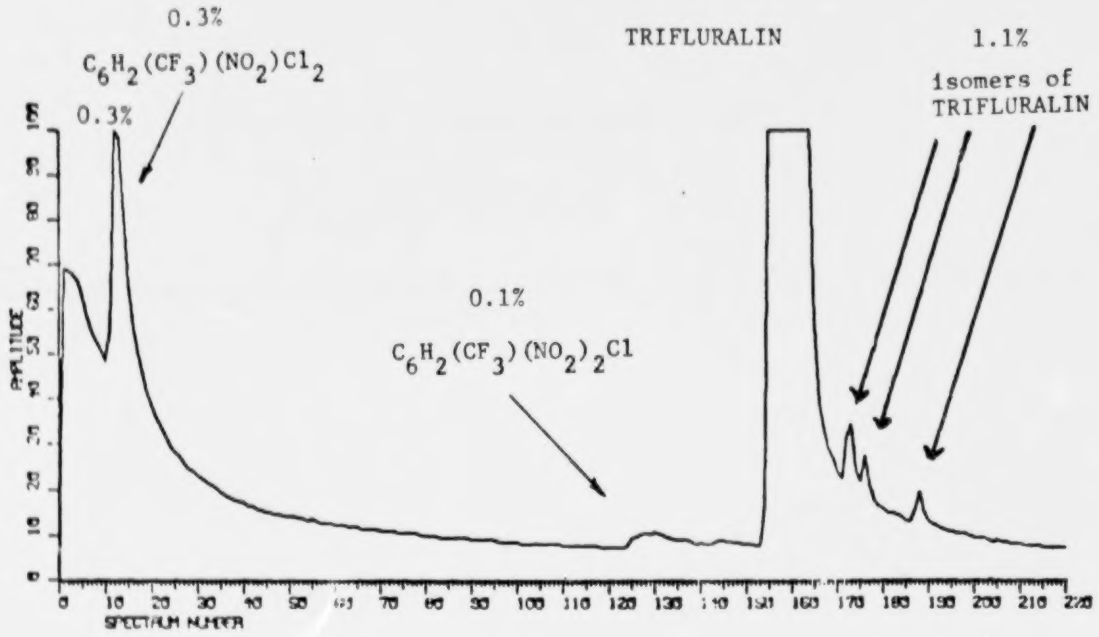

FIGURE 8. GC-MS OF TECHNICAL-GRADE TRIFLURALIN

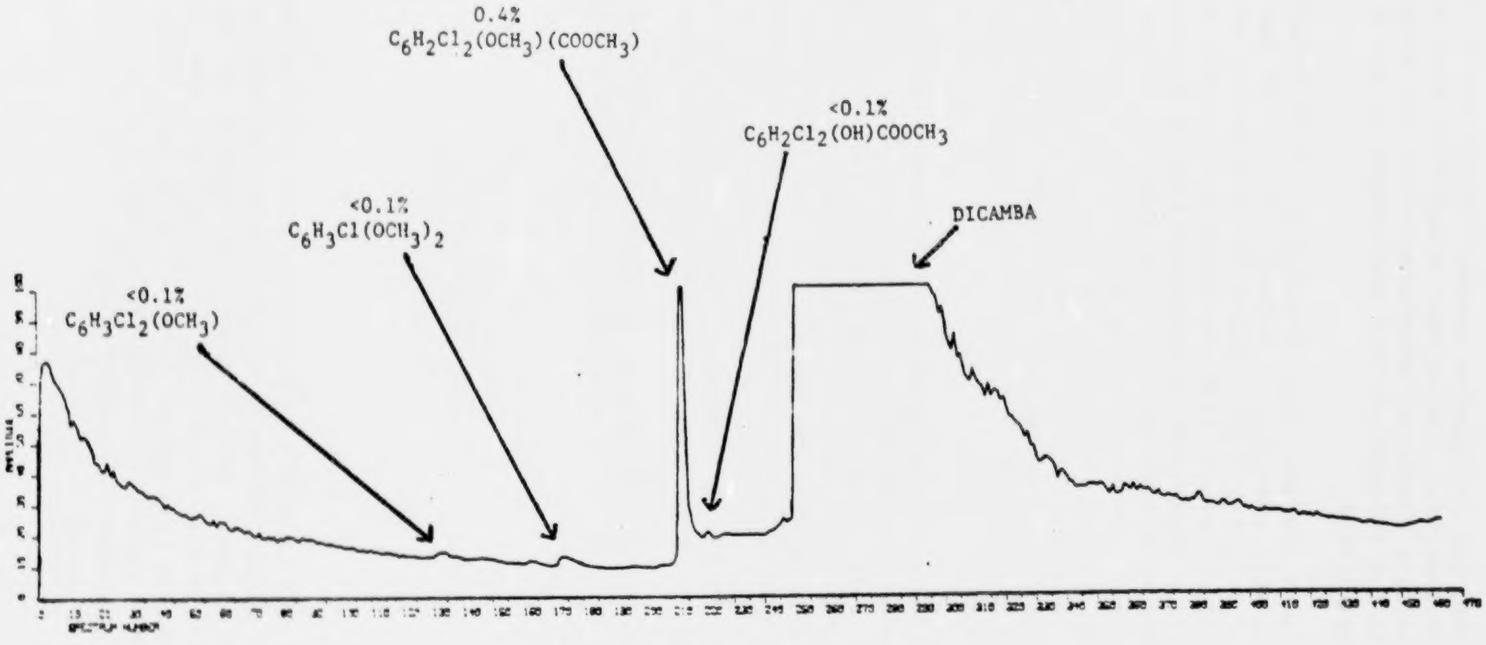

FIGURE 9. GC-MS OF TECHNICAL-GRADE DICAMBA

initially for dissolving the hydrocarbon standard variations in the concentration of the standard resulting from solvent evaporation were avoided. The use of carbon disulfide minimized interference from the solvent peak. The use of the more dilute solution avoided overloading of the GC column and minimized ghosting effects.

In order to achieve adequate equilibration of the GC injector and column and thus obtain good quantitative values, numerous GC runs were made. For each set of runs for a given pesticide a blank was run initially to make sure that no interferences from the previously assayed material were present. Successive injections of reference samples alternated with technical-grade samples were then made until the response levels became reproducible. Triplicate runs of reference and technical-grade samples were then made and the average values for the three used for calculating the product purity. In some cases triplicate runs of reference and technical-grade samples were required to accommodate the column before reproducible response levels could be obtained. In such cases a dozen GC runs were required for assaying one pesticide.

Twenty-four of the technical-grade pesticides were assayed for purity on two separate occasions using the above procedure. The results are given in Table 2. The values obtained from the two assays were the same within experimental error for all of the materials except acephate, captan, and folpet. The values also agree quite well with those stated by the manufacturer. Acephate and captan appear to have degraded somewhat but considerable more work would be needed to prove that this is indeed the case. The second assay for folpet was significantly higher than the first indicating that the reference sample had degraded somewhat.*

Any further studies on degradation rates should be carried out at various temperatures of 0 to 50 °C using reference samples stored at a very low temperature.

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* The standard solutions as well as the solutions of technical-grade material were used within two weeks of preparation and were stored at 0 °C except during use. The pure reference samples and technical-grade materials were stored at -20 °C. It is unlikely that any significant degradation occurred at -20 °C. It is more likely that the differences found resulted from decomposition of solutions, solution preparation errors, or nonhomogeneity of samples.

APPENDIX A

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APPENDIX B

REACTIONS AND POTENTIAL IMPURITIES

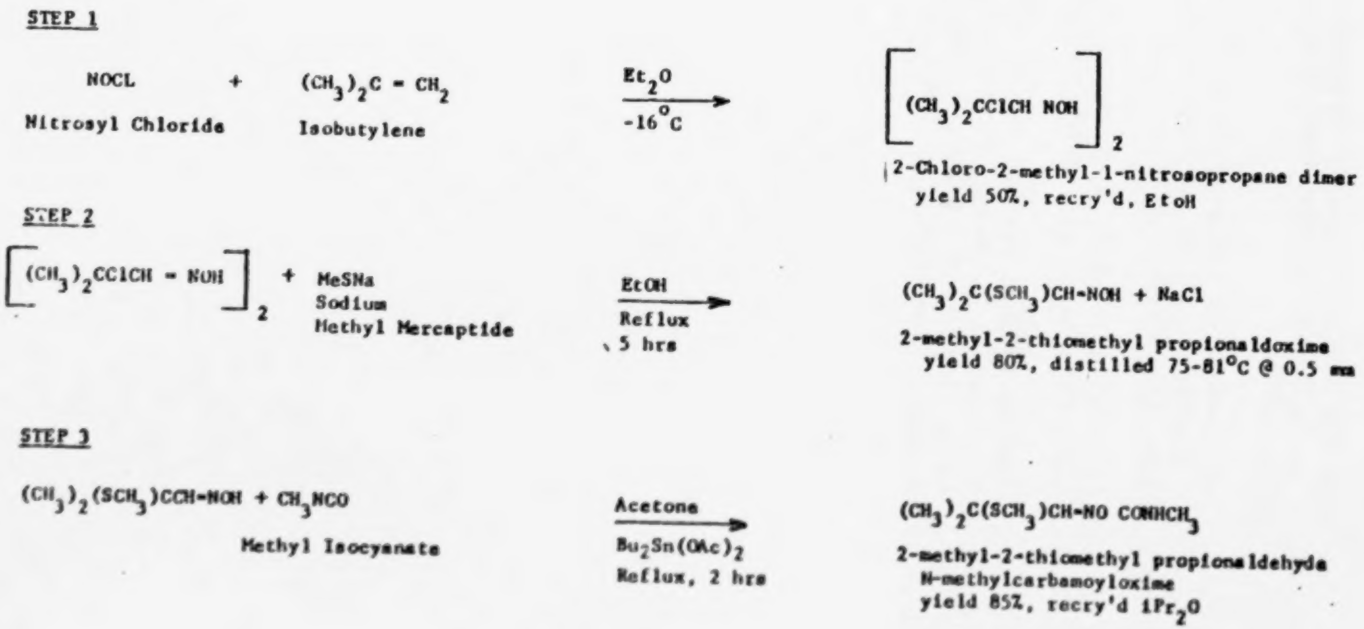

STEP 1
NOCL
+
$(CH_3)_2C = CH_2$
Nitrosyl Chloride
Isobutylene
Et_2O
$-16^{o}C$
$[(CH_3)_2CClCH\ NOH]_2$
2-Chloro-2-methyl-1-nitrosopropane dimer
yield 50%, recry'd, EtoH
STEP 2
$[(CH_3)_2CClCH = NOH]_2$
+
MeSNa
Sodium
Methyl Mercaptide
EtOH
Reflux
5 hrs
$(CH_3)_2C(SCH_3)CH\text{-}NOH + NaCl$
2-methyl-2-thiomethyl propionaldoxime
yield 80%, distilled 75-81°C @ 0.5 mm
STEP 3
$(CH_3)_2(SCH_3)CCH\text{-}NOH + CH_3NCO$
Methyl Isocyanate
Acetone
$Bu_2Sn(OAc)_2$
Reflux, 2 hrs
$(CH_3)_2C(SCH_3)CH\text{-}NO\ CONHCH_3$
2-methyl-2-thiomethyl propionaldehyde
N-methylcarbamoyloxime
yield 85%, recry'd iPr_2O

FIGURE B-1. SYNTHESIS OF ALDICARB

TABLE B-1. REACTIONS OCCURRING DURING ALDICARB PRODUCTION

Reaction Number	Generalized Reaction
1	$R_2C{=}CH_2 + NOCl \rightarrow R_2CClCH_2NO \rightleftharpoons R_2CClCH{=}NOH \rightleftharpoons [R_2CClCH{=}NOH]_2$
2	$R_2C{=}CH_2 + NOCl \rightarrow R_2C(NO)CH_2Cl$
3	$RNO \xrightarrow[NOCl]{[O]} RNO_2$
4	$RCH{=}NOH \xrightarrow[H^+]{H_2O} RCHO$
5	$RCHO \xrightarrow{[O]} RCOOH$
6	$RCl + CH_3SNa \rightarrow RSCH_3$
7	$RCl \xrightarrow{C_2H_5O^-} ROC_2H_5$
8	$RCl \xrightarrow{OH^-} ROH$
9	$RCl + ROH \xrightarrow{OH^-} ROR$
10	$RCH{=}NOH + RCl \rightarrow RCH{=}NOR$
11	$RCH{=}NOH + RCl \rightarrow RCH{=}NRO$
12	$RSCH_3 \xrightarrow{[O]} RSOCH_3$
13	$RSOCH_3 \xrightarrow{[O]} RSO_2CH_3$
14	$RCH{=}NOH + CH_3NCO \rightarrow RCH{=}NOCONHCH_3$
15	$RCH{=}NOH + CH_3NCO \rightarrow RCN + CH_3NH_2 + CO_2$
16	$CH_3NCO \xrightarrow{H_2O} CH_3NH_2 + CO_2$
17	$CH_3NH_2 + CH_3NCO \rightarrow CH_3NHCONHCH_3$
18	$R{-}OH + CH_3NCO \rightarrow ROCONHCH_3$
19	$RCOOH + CH_3NCO \rightarrow [RCOOCONHCH_3] \rightarrow RCONHCH_3 + CO_2$

TABLE B-1. (Continued)

Reaction Number	Generalized Reaction
20	$RCH{=}NOCONHCH_3 + CH_3NCO \rightarrow RCH{=}NOCON(CONHCH_3)CH_3$
21	$RNHCONHCH_3 + CH_3NCO \rightarrow RNHCON(CONHCH_3)CH_3$
22	$RCHO + CH_3COCH_3 \xrightarrow[OH^-]{H^+} RCH{=}CHCOCH_3$
23	$Bu_2Sn(OAc)_2 \xrightarrow{H_2O} Bu_2SnO$

TABLE B-2. POSSIBLE CONTAMINANTS IN ALDICARB

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
1	$(CH_3)_2C{=}CH_2$	SM	--	1	0
2	$(CH_3)_2CClCH{=}NOH$	1	1	1	+
3	$(CH_3C(NO)CH_2Cl$	1	2	1	0
4	$(CH_3)_2CClCH_2NO_2$	2	3	1	0
5	$(CH_3)_2C(NO_2)CH_2Cl$	3	3	1	0
6	$(CH_3)_2CClCHO$	2	4	1	0
7	$(CH_3)_2CClCOOH$	6	5	1	0
8	$(CH_3)_2C(SCH_3)CH{=}NOH$	2	6	2	++
9	$(CH_3)_2C(NO)CH_2SCH_3$	3	6	2	+
10	$(CH_3)_2C(SCH_3)CH_2NO_2$	4	6	2	+
11	$(CH_3)_2C(NO_2)CH_2SCH_3$	5	6	2	+
12	$(CH_3)_2C(SCH_3)CHO$	6	6	2	0
13	$(CH_3)_2C(SCH_3)COOH$	7 12	6 5	2 2 or St	0
14	$(CH_3)_2C(OC_2H_5)CH{=}NOH$	2	7	2	+
15	$(CH_3)_2C(NO)CH_2OC_2H_5$	3	7	2	0
16	$(CH_3)_2C(OC_2H_5)CH_2NO_2$	4	7	2	0
17	$(CH_3)_2C(NO_2)CH_2OC_2H_5$	5	7	2	0
18	$(CH_3)_2C(OC_2H_5)CHO$	6	7	2	0
19	$(CH_3)_2C(OC_2H_5)COOH$	7 18	7 5	2 2 or St	0
20	$(CH_3)_2C(OH)CH{=}NOH$	2	8	2	0
21	$(CH_3)_2C(NO)CH_2OH$	3	8	2	0

TABLE B-2. (Continued)

No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[a]	Likelihood of Being Present in Final Product
22	$(CH_3)_2C(OH)CH_2NO_2$	4	8	2	0
23	$(CH_3)_2C(NO_2)CH_2OH$	5	8	2	0
24	$(CH_3)_2C(OH)CHO$	6	8	2	0
25	$(CH_3)_2C(OH)COOH$	7 24	8 5	2 2 or St	0
26	$(CH_3)_2\ C[OC(CH_3)_2CH{=}NOH]CH{=}NOH$	2 + 20	9	2	0
27	$(CH_3)_2\ C[OCH_2C(NO)(CH_3)_2]CH{=}NOH$	2 + 21 3 + 20	9 9	2 2	0
28	$(CH_3)_2\ C[OC(CH_3)_2CH_2NO_2]\ CH{=}NOH$	2 + 22 4 + 20	9 9	2 2	0
29	$(CH_3)_2C[OCH_2C(NO_2)(CH_3)_2]CH{=}NOH$	2 + 23 5 + 20	9 9	2 2	0
30	$(CH_3)_2C[OC(CH_3)_2CHO]CH{=}NOH$	2 + 24 6 + 20	9 9	2 2	0
31	$(CH_3)_2C[OC(CH_3)_2COOH]CH{=}NOH$	2 + 25 7 + 20 30	9 9 5	2 2 2 or St	0
32	$(CH_3)_2C(NO)CH_2OCH_2C(NO)(CH_3)_2$	3 + 21	9	2	0
33	$(CH_3)_2C(NO)CH_2OC(CH_3)_2CH_2NO_2$	3 + 22 4 + 21	9 9	2 2	0
34	$(CH_3)_2C(NO)CH_2OCH_2C(NO_2)(CH_3)_2$	[illegible] + 23 5 + 21	9 9	2 2	0
35	$(CH_3)_2C(NO)CH_2OC(CH_3)_2CHO$	3 + 24 6 + 21	9 9	2 2	0
36	$(CH_3)_2C(NO)CH_2OC(CH_3)_2COOH$	3 + 25 7 + 21 35	9 9 5	2 2 2 or St	0
37	$(CH_3)_2C[OC(CH_3)_2CH_2NO_2]CH_2NO_2$	4 + 22	9	2	0
38	$(CH_3)_2C[OCH_2C(NO_2)(CH_3)_2]CH_2NO_2$	4 + 23 5 + 22	9 9	2 2	0
39	$(CH_3)_2C[OC(CH_3)_2CHO]CH_2NO_2$	4 + 24 6 + 22	9 9	2 2	0

TABLE B-2. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
40	$(CH_3)_2C[OC(CH_3)_2COOH]CH_2NO_2$	4 + 25 7 + 22 39	9 9 5	2 2 2 or St	0
41	$(CH_3)_2C(NO_2)CH_2OCH_2C(NO_2)(CH_3)_2$	5 + 23	9	2	0
42	$(CH_3)_2C(NO_2)CH_2OC(CH_3)_2CHO$	5 + 24 6 + 23	9 9	2 2	0
43	$(CH_3)_2C(NO_2)CH_2OC(CH_3)_2COOH$	5 + 25 7 + 23 42	9 9 5	2 2 2 or St	0
44	$(CH_3)_2C[OC(CH_3)_2CHO]CHO$	6 + 24	9	2	0
45	$(CH_3)_2C[OC(CH_3)_2COOH]CHO$	6 + 25 7 + 24 44	9 9 5	2 2 2 or St	0
46	$(CH_3)_2C[OC(CH_3)_2COOH]COOH$	7 + 25 45	9 5	2 2 or St	0
47	$(CH_3)_2CClCH{=}NOC(CH_3)_2CH{=}NOH$	2 + 2	10	2	+
48	$(CH_3)_2CClCH{=}NOCH_2C(NO)(CH_3)_2$	2 + 3	10	2	0
49	$(CH_3)_2CClCH{=}NOC(CH_3)_2CH_2NO_2$	2 + 4	10	2	0
50	$(CH_3)_2CClCH{=}NOCH_2C(NO_2)(CH_3)_2$	2 + 5	10	2	0
51	$(CH_3)_2CClCH{=}NOC(CH_3)_2CHO$	2 + 6	10	2	0
52	$(CH_3)_2CClCH{=}NOC(CH_3)_2COOH$	2 + 7 51	10 5	2 2 or St	0
53	$(CH_3)_2C(SCH_3)CH{=}NOC(CH_3)_2CH{=}NOH$	8 + 2	10	2	+
54	$(CH_3)_2C(SCH_3)CH{=}NOCH_2C(NO)(CH_3)_2$	8 + 3	10	2	0
55	$(CH_3)_2C(SCH_3)CH{=}NOC(CH_3)_2CH_2NO_2$	8 + 4	10	2	0
56	$(CH_3)_2C(SCH_3)CH{=}NOCH_2C(NO_2)(CH_3)_2$	8 + 5	10	2	0
57	$(CH_3)_2C(SCH_3)CH{=}NOC(CH_3)_2CHO$	8 + 6	10	2	0
58	$(CH_3)_2C(SCH_3)CH{=}NOC(CH_3)COOH$	8 + 7 57	10 5	2 2 or St	0
59	$(CH_3)_2C(OC_2H_5)CH{=}NOC(CH_3)_2CH{=}NOH$	14 + 2	10	2	0
60	$(CH_3)_2C(OC_2H_5)CH{=}NOCH_2C(NO)(CH_3)_2$	14 + 3	10	2	0

TABLE B-2 (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
61	$(CH_3)_2C(OC_2H_5)CH{=}NOC(CH_3)_2CH_2NO_2$	14 + 4	10	2	0
62	$(CH_3)_2C(OC_2H_5)CH{=}NOCH_2C(NO_2)CH_3)_2$	14 + 5	10	2	0
63	$(CH_3)_2C(OC_2H_5)CH{=}NOC(CH_3)_2CHO$	14 + 6	10	2	0
64	$(CH_3)_2C(OC_2H_5)CH{=}NOC(CH_3)_2COOH$	14 + 7	10	2	0
65	$(CH_3)_2C(OH)CH{=}NOC(CH_3)_2CH{=}NOH$	20 + 2	10	2	0
66	$(CH_3)_2C(OH)CH{=}NOCH_2C(NO)(CH_3)_2$	20 + 3	10	2	0
67	$(CH_3)_2C(OH)CH{=}NOC(CH_3)_2CH_2NO_2$	20 + 4	10	2	0
68	$(CH_3)_2C(OH)CH{=}NOCH_2C(NO_2)(CH_3)_2$	20 + 5	10	2	0
69	$(CH_3)_2C(OH)CH{=}NOC(CH_3)_2CHO$	20 + 6	10	2	0
70	$(CH_3)_2C(OH){=}NOC(CH_3)_2COOH$	20 + 7 69	10 5	2 2 or St	0
71	$(CH_3)_2CClCH{=}N(O)C(CH_3)_2CH{=}NOH$	2 + 2	11	2	+
72	$(CH_3)_2CClCH{=}N(O)CH_2C(NO)(CH_3)_2$	2 + 3	11	2	0
73	$(CH_3)_2CClCH{=}N(O)C(CH_3)_2CH_2NO_2$	2 + 4	11	2	0
74	$(CH_3)_2CClCH{=}N(O)CH_2C(NO_2)(CH_3)_2$	2 + 5	11	2	0
75	$(CH_3)_2CClCH{=}N(O)C(CH_3)_2CHO$	2 + 6	11	2	0
76	$(CH_3)_2CClCH{=}N(O)C(CH_3)_2COOH$	2 + 7 75	11 5	2 2 or St	0
77	$(CH_3)_2C(SCH_3)CH{=}N(O)C(CH_3)_2CH{=}NOH$	8 + 2	11	2	+
78	$(CH_3)_2C(SCH_3)CH{=}N(O)CH_2C(NO)(CH_3)_2$	8 + 3	11	2	0
79	$(CH_3)_2C(SCH_3)CH{=}N(O)C(CH_3)_2CH_2NO_2$	8 + 4	11	2	0
80	$(CH_3)_2C(SCH_3)CH{=}N(O)CH_2C(NO_2)(CH_3)_2$	8 + 5	11	2	0
81	$(CH_3)_2C(SCH_3)CH{=}N(O)C(CH_3)_2CHO$	8 + 6	11	2	0
82	$(CH_3)_2C(SCH_3)CH{=}N(O)C(CH_3)COOH$	8 + 7 8	11 5	2 2 or St	0
83	$(CH_3)_2C(OC_2H_5)CH{=}N(O)C(CH_3)_2CH{=}NOH$	14 + 2	11	2	0

TABLE B-2. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
84	$(CH_3)_2C(OC_2H_5)CH{=}N(O)CH_2C(NO)(CH_3)_2$	14 + 3	11	2	0
85	$(CH_3)_2C(OC_2H_5)CH{=}N(O)C(CH_3)_2CH_2CH_2NO_2$	14 + 4	11	2	0
86	$(CH_3)_2C(OC_2H_5)CH{=}N(O)CH_2C(NO_2)(CH_3)_2$	14 + 5	11	2	0
87	$(CH_3)_2C(OC_2H_5)CH{=}N(O)C(CH_3)_2CHO$	14 + 6	11	2	0
88	$(CH_3)_2C(OC_2H_5)CH{=}N(O)C(CH_3)_2COOH$	14 + 7 87	11 5	2 2 or St	0
89	$(CH_3)_2C(OH)CH{=}N(O)C(CH_3)_2CH{=}NOH$	20 + 2	11	2	0
90	$(CH_3)_2C(OH)CH{=}N(O)CH_2C(NO)(CH_3)_2$	20 + 3	11	2	0
91	$(CH_3)_2C(OH)CH{=}N(O)C(CH_3)_2CH_2CH_2CH_2NO_2$	20 + 4	11	2	0
92	$(CH_3)_2C(OH)CH{=}N(O)CH_2C(NO_2)(CH_3)_2$	20 + 5	11	2	0
93	$(CH_3)_2C(OH)CH{=}N(O)C(CH_3)_2CHO$	20 + 6	11	2	0
94	$(CH_3)_2C(OH)CH{=}N(O)C(CH_3)_2COOH$	20 + 7 93	11 5	2 2 or St	0
95	$(CH_3)_2C(SOCH_3)CH{=}NOH$	8	12	2 or St	+
96	$(CH_3)_2C(NO)CH_2SOCH_3$	9	12	2 or St	0
97	$(CH_3)_2C(SOCH_3)CH_2NO_2$	10	12	2 or St	0
98	$(CH_3)_2C(NO_2)CH_2SOCH_3$	11	12	2 or St	0
99	$(CH_3)_2C(SOCH_3)CHO$	12	12	2 or St	0
100	$(CH_3)_2C(SOCH_3)COOH$	13 99	12 5	2 or St 2 or St	0
101	$(CH_3)_2C(SO_2CH_3)CH{=}NOH$	8	13	2 or St	+
102	$(CH_3)_2C(NO)CH_2SO_2CH_3$	9	13	2 or St	0
103	$(CH_3)_2C(SO_2CH_3)CH_2NO_2$	10	13	2 or St	0
104	$(CH_3)_2C(NO_2)CH_2SO_2CH_3$	11	13	2 or St	0
105	$(CH_3)_2C(SO_2CH_3)CHO$	12	13	2 or St	0
106	$(CH_3)_2C(SO_2CH_3)COOH$	13	13 5	2 or St	0

TABLE B-2. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
107	$(CH_3)_2CClCH=NOCONHCH_3$	2	14	3	+
108	$(CH_3)_2C(SCH_3)CH=NOCONHCH_3$	8	14	3	Aldicarb
109	$(CH_3)_2C(OC_2H_5)CH=NOCONHCH_3$	14	14	3	++
110	$(CH_3)_2C(OH)CH=NOCONHCH_3$	20	14	3	0
111	$(CH_3)_2C[OC(CH_3)_2CH=NOH]CH=NOCONHCH_3$	26	14	3	0
112	$(CH_3)_2C[OC(CH_3)_2CH=NOCONHCH_3]$ $CH=NOCONHCH_3$	111	14	3	+
113	$(CH_3)_2C[OCH_2C(NO)(CH_3)_2]CH=NOCONHCH_3$	27	14	3	0
114	$(CH_3)_2C[OC(CH_3)_2CH_2NO_2]CH=NOCONHCH_3$	28	14	3	0
115	$(CH_3)_2C[OCH_2C(NO_2)(CH_3)_2]CH=NOCONHCH_3$	29	14	3	0
116	$(CH_3)_2C[OC(CH_3)_2CHO]CH=NOCONHCH_3$	30	14	3	0
117	$(CH_3)_2C[OC(CH_3)_2COOH]CH=NOCONHCH_3$	31 116	14 5	3 St	0
118	$(CH_3)_2CClCH=NOC(CH_3)_2CH=NOCONHCH_3$	47	14	3	0
119	$(CH_3)_2C(SCH_3)CH=NOC(CH_3)_2CH=NOCONHCH_3$	53	14	3	+
120	$(CH_3)_2C(SOCH_3)CH=NOC(CH_3)_2CH=NOCONHCH_3$	119	12	St	0
121	$(CH_3)_2C(SO_2CH_3)CH=NOC(CH_3)_2$ $CH=NOCONHCH_3$	120	13	St	0
122	$(CH_3)_2C(OC_2H_5)CH=NOC(CH_3)_2CH=NOCONHCH_3$	59	14	3	0
123	$(CH_3)_2C(OH)CH=NOC(CH_3)_2CH=NOCONHCH_3$	65	14	3	0
124	$(CH_3)_2CClCH=N(O)C(CH_3)_2CH=NOCONHCH_3$	71	14	3	0
125	$(CH_3)_2C(SCH_3)CH=N(O)C(CH_3)_2$ $CH=NOCONHCH_3$	77	14	3	+
126	$(CH_3)_2C(SOCH_3)CH=N(O)C(CH_3)_2$ $CH=NOCONHCH_3$	125	12	St	0
127	$(CH_3)C(SO_2CH_3)CH=N(O)C(CH_3)_2$ $CH=NOCONHCH_3$	126	13	St	0

TABLE B-2. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
128	$(CH_3)_2C(OC_2H_5)CH=N(O)C(CH_3)_2$ $CH=NOCONHCH_3$	83	14	3	0
129	$(CH_3)_2C(OH)CH=N(O)C(CH_3)_2$ $CH=NOCONHCH_3$	89	14	3	0
130	$(CH_3)_2C(SOCH_3)CH=NOCONHCH_3$	108	12	St	++
131	$(CH_3)_2C(SO_2CH_3)CH=NOCONHCH_3$	130	13	St	+
132	$(CH_3)_2CClCN$	2	15	3	0
133	$(CH_3)_2C(SCH_3)CN$	8	15	3	++
134	$(CH_3)_2C(OC_2H_5)CN$	14	15	3	+
135	$(CH_3)_2C(OH)CN$	20	15	2	0
136	$(CH_3)_2C(SOCH_3)CN$	133	12	St	+
137	$(CH_3)_2C(SO_2CH_3)CN$	136	13	St	0
138	$(CH_3NCO$	SM	--	3	0
139	CH_3NH_2	138	15 or 16	3	+
140	$CH_3NHCONHCH_3$	139	17	3	++
141	$CH_3NHCON(CONHCH_3)CH_3$	140	21	3	++
142	$(CH_3)_2C(OCONHCH_3)CH=NOH$	20	18	3	0
143	$(CH_3)_2C(NO)CH_2OCONHCH_3$	21	18	3	0
144	$(CH_3)_2C(OCONHCH_3)CH_2NO_2$	22	18	3	0
145	$(CH_3)_2C(NO_2)CH_2OCONHCH_3$	23	18	3	0
146	$(CH_3)_2C(OCONHCH_3)CHO$	24	18	3	0
147	$(CH_3)_2C(OCONHCH_3)COOH$	25 146	18 5	3 St	0
148	$(CH_3)_2C(OCONHCH_3)CH=NOCONHCH_3$	110 142	18 14	3 3	+
149	$(CH_3)_2CClCONHCH_3$	7	19	3	0

TABLE B-3. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product
150	$(CH_3)_2C(SCH_3)CONHCH_3$	13	19	3	+
151	$(CH_3)_2C(OC_2H_5)CONHCH_3$	19	19	3	+
152	$(CH_3)_2C(OH)CONHCH_3$	25	19	3	0
153	$(CH_3)_2C(SOCH_3)CONHCH_3$	100	19	3	+
154	$(CH_3)_2C(SO_2CH_3)CONHCH_3$	106 153	19 13	3 St	0
155	$(CH_3)_2C(OCONHCH_3)CONHCH_3$	147 152	19 18	3 3	+
156	$(CH_3)_2C(SCH_3)CH{=}NOCON(CONHCH_3)CH_3$	108	20	3	++
157	$(CH_3)_2C(SOCH_3)CH{=}NOCON(CONHCH_3)CH_3$	156	12	St	+
158	$(CH_3)_2C(SO_2CH_3)CH{=}NOCON(CONHCH_3)CH_3$	157	13	St	0
159	$(CH_3)_2CCLCH{=}CHCOCH_3$	6	22	3	0
160	$(CH_3)_2C(SCH_3)CH{=}CHCOCH_3$	12	22	3	0
161	$(CH_3)_2C(OC_2H_5)CH{=}CHCOCH_3$	18	22	3	0
162	$(CH_3)_2C(OH)CH{=}CHCOCH_3$	24	22	3	0
163	$(CH_3)_2C(OCONHCH_3)CH{=}CHCOCH_3$	146 162	22 18	3 3	
164	$(CH_3)_2C(SOCH_3)CH{=}CHCOCH_3$	160	12	St	0
165	$(CH_3)_2C(SO_2CH_3)CH{=}CHCOCH_3$	164	13	St	0
166	$Bu_2Sn(OAc)_2$	Cat.	--	--	+
167	Bu_2SnO	166	23	St	+

(a) Precursor refers to the contaminant numbered in this table; SM = Starting material; Cat. = catalyst.

(b) See Table B-1.

(c) The step (see Figure B-1) during which the contaminant might be formed; St indicates possible formation during storage.

(d) 0 = likely present only at levels below 1 ppm
+ = likely present at levels above 1 ppm but below 100 ppm.
++ = likely present at levels above 100 ppm.

Step 1 $$4C_3H_7OH + P_2S_5 \longrightarrow 2(C_3H_7O)_2P(S)SH + H_2S$$

O, O-di-n-propyl
phosphorodithioate

Step 2 $$(C_3H_7O)_2P(S)SH + Cl_2 \longrightarrow (C_3H_7O)_2P(S)Cl + HCl$$

O, O-di-n-propyl
chlorophosphorothioate

Step 3 $$2(C_3H_7O)_2P(S)Cl + Na_2CO_3 \xrightarrow[H_2O]{pyridine} [(C_3H_7O)_2P(S)]_2O + CO_2 + 2NaCl$$

ASPON

FIGURE B-2. SYNTHESIS OF ASPON

TABLE B-3. REACTIONS OCCURRING DURING PRODUCTION OF ASPON

Number	Generalized Reaction[a]
1	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6	$(RO)_2P(Y)SH \xrightarrow{P_2S_5} (RO)(RS)P(Y)SH + (RS)_2P(Y)SH$
7	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
8	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$
9	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
10	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)Y\ P(Y)(RY)_2 + H_2Y$
11	$2(RY)_2\ P(Y)SH \xrightarrow{[O]} (RY)_2\ P(Y)\ S\ S\ P(Y)(RY)_2$
12	$2(RY)_2P(Y)SH + Cl_2 \longrightarrow (RY)_2P(Y)SS\ P(Y)(RY)_2 + 2HCl$
13	$(RY)_2\ P(S)SH \longrightarrow (RY)_2PSH + S$
14	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
15	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$

TABLE B-3. (Continued)

Number	Generalized Reaction(a)
16	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)S\,P(RY)_2$
17	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2\,P(O)SR'$
18	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
19	$(RY)_2P(S)SH + Cl_2 \longrightarrow (RY)_2P(S)Cl + S_2Cl_2$
20	$(RO)_2P(S)Cl + C_5H_5N \longrightarrow [(RO)_2\,P(S)NC_5H_5]^+Cl^-$
21	$[(RO)_2P(S)NC_5H_5]^+Cl^- \longrightarrow RCl + C_5H_5N + \text{Polymer}$
22	$2(RO)_2\,P(S)Cl + H_2O \xrightarrow[Na_2CO_3]{C_5H_5N} [(RO)_2P(S)]_2O$
23	$[(RO)_2P(S)]_2O + H_2O \longrightarrow 2(RO)_2\,P(S)OH$
24	$(RO)_2P(S)OH + H_2O \longrightarrow RO\,P(S)(OH)_2 + ROH$
25	$RO\,P(S)(OH)_2 + 2H_2O \longrightarrow ROH + H_3PO_4 + H_2S$

(a) R and R' = alkyl or substituted alkyl.
Y = oxygen or sulfur.

TABLE B-4. POSSIBLE CONTAMINANTS IN ASPON

Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Probability of Being Present in Final Product[d]
1	P_2S_5	SM	–	1	0
2	$CH_3CH_2CH_2OH$	SM 39	– 23-25	1 St	*
3	$CH_3CHOHCH_3$	Im-2	–	1	0
4	CH_3CH_2OH	Im-2	–	1	0
5	$CH_3CH_2CH_2CH_2OH$	Im-2	–	1	0
6	$(CH_3CH_2CH_2O)_2P(S)SH$	1+2	1	1	*
7	$[(CH_3)_2CHO]_2P(S)SH$	1+3	1	1	0
8	$(CH_3CH_2O)_2\ P(S)SH$	1+4	1	1	0
9	$(n\text{-}C_4H_9O)_2\ P(S)SH$	1+5	1	1	0
10	$(n\text{-}C_3H_7O)_2(n\text{-}C_3H_7S)PS$	2+6	2	1	**
11	$(n\text{-}C_3H_7O)_3\ PS$	2+6 2+10	3 4	1 1	**
12	$(n\text{-}C_3H_7O)(n\text{-}C_3H_7S)_2\ PS$	1+10	5	1	*
13	$(n\text{-}C_3H_7O)(n\text{-}C_3H_7S)P(S)SH$	6	6	1	*
14	$(n\text{-}C_3H_7O)_2\ P(S)OH$	6 11 39	7 8 23	1 1 St	*
15	$(n\text{-}C_3H_7O)_2\ P(O)SH$	14	9	1	0
16	$(n\text{-}C_3H_7O)_2\ P(O)OH$	15	7	1	0
17	$(n\text{-}C_3H_7O)(n\text{-}C_3H_7S)P(O)SH$	12	8+9	1	0
18	$(n\text{-}C_3H_7S)_2\ P(S)SH$	6	6	1	0

TABLE B-4. (Continued)

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Probability of Being Present in Final Product(d)
19	$(n\text{-}C_3H_7O)_2\,P(S)S\,P(S)(n\text{-}C_3H_7O)_2$	6	10	1	+
20	$(n\text{-}C_3H_7O)_2\,P(S)\,S\,S\,P(S)(n\text{-}C_3H_7O)_2$	6 6	11 12	1 2	++
21	$(n\text{-}C_3H_7O)_2\,PSH$	6	13	1	0
22	$(n\text{-}C_3H_7O)_3\,P$	21	14	1	0
23	$(n\text{-}C_3H_7O)_2(n\text{-}C_3H_7S)\,P$	21	15	1	0
24	$(n\text{-}C_3H_7O)_2\,P(S)\,S\,P(n\text{-}C_3H_7O)_2$	21	16	1	0
25	$(n\text{-}C_3H_7O)_2(n\text{-}C_3H_7S)\,PO$	11	17	1	+
26	$(n\text{-}C_3H_7O)_3\,PO$	2+25	4	1	0
27	$CH_3CH_2CH_2SH$	2+10	4	1	0
28	$(n\text{-}C_3H_7S)_3\,PS$	12+27	4	1	0
29	$(n\text{-}C_3H_7S)_3\,PO$	28	18	1	0
30	Cl_2	SM	–	2	0
31	$(n\text{-}C_3H_7O)_2\,P(S)Cl$	6+30	19	2	++
32	$(CH_3CH_2O)_2\,P(S)Cl$	8+30	19	2	0
33	$[(CH_3)_2CHO]_2\,P(S)Cl$	7+30	19	2	0
34	$(n\text{-}C_4H_9O)_2\,P(S)Cl$	9+30	19	2	0
35	C_5H_5N	Cat.	–	3	+
36	$[(n\text{-}C_3H_7O)_2\,P(S)\,NC_5H_5]^+Cl^-$	31+35	20	3	+

TABLE B-4. (Continued)

Number	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Probability of Being Present in Final Product[(d)]
37	$CH_3CH_2CH_2Cl$	36	21	3	0
38	Polymer (containing H, C, O, P and S)	36	21	3	++
39	$[(n\text{-}C_3H_7O)_2 P(S)]_2 O$	31	22	3	ASPON
40	$(n\text{-}C_3H_7O)(n\text{-}C_3H_7S) P(O) OP(S)(n\text{-}C_3H_7O)_2$	39	17	3 or St	++
41	$[(n\text{-}C_3H_7O)(n\text{-}C_3H_7S) P(O)]_2 O$	40	17	3 or St	++
42	$[(C_2H_5O)_2 P(S)]_2 O$	32	22	3	+
43	$[(CH_3)_2CHO]_2 P(S) O P(S)[OCH(CH_3)_2]_2$	33	22	3	+
44	$[(n\text{-}C_4H_9O)_2 P(S)]_2 O$	34	22	3	+
45	$n\text{-}C_3H_7O P(S)(OH)_2$	14	24	St	+
46	H_3PO_4	39	23-25	St	+
47	H_2S	39	23-25	St	+

(a) Precursor refers to the contaminant numbered in this table
SM = starting material
Cat = catalyst
Im-2 = impurity in No. 2.

(b) See Table B-3.

(c) The step (Figure B-2) during which the contaminant might be formed.
St indicates possible formation during storage.

(d) 0 = probably present only at levels below 1 ppm.
+ = probably present at levels above 1 ppm but below 100 ppm
++ = probably present at levels above 100 ppm.

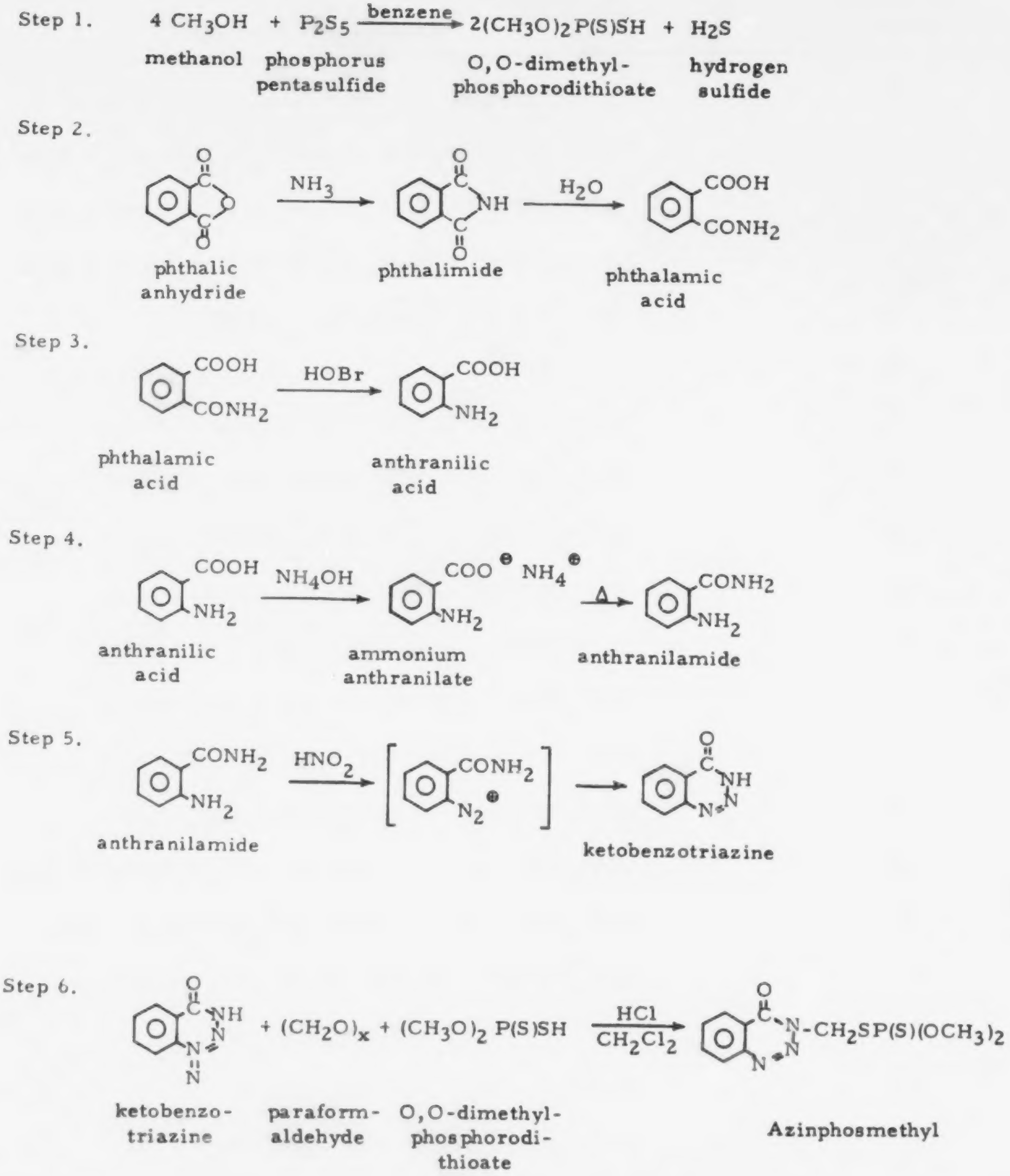

Step 1.
4 CH3OH + P2S5 —benzene→ 2(CH3O)2P(S)SH + H2S
methanol
phosphorus pentasulfide
O,O-dimethyl-phosphorodithioate
hydrogen sulfide
Step 2.
NH3
H2O
COOH
CONH2
phthalic anhydride
phthalimide
phthalamic acid
Step 3.
HOBr
COOH
CONH2
NH2
phthalamic acid
anthranilic acid
Step 4.
NH4OH
Δ
COOH
NH2
COO⊖ NH4⊕
CONH2
anthranilic acid
ammonium anthranilate
anthranilamide
Step 5.
HNO2
CONH2
NH2
N2⊕
anthranilamide
ketobenzotriazine
Step 6.
+ (CH2O)x + (CH3O)2 P(S)SH
HCl
CH2Cl2
N-CH2SP(S)(OCH3)2
ketobenzo-triazine
paraform-aldehyde
O,O-dimethyl-phosphorodi-thioate
Azinphosmethyl

FIGURE B-3. MAIN REACTIONS IN THE PREPARATION OF AZINPHOSMETHYL

TABLE B-5. REACTIONS OCCURRING DURING PRODUCTION OF AZINPHOSMETHYL

Reaction Number	Generalized Reaction(a)
1.	$4\ ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2.	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3.	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4.	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5.	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6.	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
7.	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
8.	$(RY)_2P(Y)OH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2O$
9.	$(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
10.	$(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
11.	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
12.	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
13.	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
14.	$(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(YR)_2$
15.	$(RY)_2P(Y)YH + R'Cl \longrightarrow (RY)_2P(Y)(YR') + HCl$
16.	$(RY)_3P(Y) + R'Cl \longrightarrow (RY)_2(R'Y)P(Y) + RCl$
17.	$(RY)_2P(Y)YH + R'_2NH \longrightarrow (RY)_2P(Y)NR'_2 + H_2Y$
18.	$(RCO)_2O + NH_3 \longrightarrow (RCO)_2NH + H_2O$
19.	$(RCO)_2O + H_2O \longrightarrow 2RCOOH$
20.	$(RCO)_2NH + NH_3 \longrightarrow 2RCONH_2$
21.	$(RCO)NH + H_2O \longrightarrow RCOOH + RCONH_2$
22.	$RCONH_2 \xrightarrow{HOBr} RNH_2 + CO_2$

TABLE B-5. (Continued)

Reaction Number	Generalized Reaction[a]
23.	$\mathrm{RCOOH \longrightarrow RCOO^{\ominus}NH_4^{\oplus} \xrightarrow{\Delta} RCONH_2}$
24.	$\mathrm{ArNH_2 \xrightarrow{HNO_2} ArN_2^{\oplus}}$
25.	$\mathrm{ArN_2^{\oplus} + ArNH_2 \longrightarrow ArN{=}NNHAr}$
26.	$\mathrm{ArN_2^{\oplus} + ArCONH_2 \longrightarrow ArN{=}NNHCOAr}$
27.	$\mathrm{ArN_2^{\oplus} \longrightarrow ArN{=}NAr}$
28.	$\mathrm{ArN_2^{\oplus} \longrightarrow ArAr}$
29.	$\mathrm{ArN_2^{\oplus} \xrightarrow{H_2O} ArOH}$
30.	$\mathrm{CH_2O + HCl \longrightarrow ClCH_2OCH_2Cl}$
31.	$\mathrm{R_2NH + CH_2O \longrightarrow R_2NCH_2OH}$
32.	$\mathrm{ArH + CH_2O \longrightarrow ArCH_2OH}$
33.	$\mathrm{ROH + HCl \longrightarrow RCl}$
34.	$\mathrm{(RY)_3PS \xrightarrow{[O]} (RY)_3PO}$
35.	$\mathrm{(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH}$
36.	$\mathrm{(RY)_2P(S)OR' \rightleftarrows (RY)_2P(O)SR'}$
37.	$\mathrm{RSH \xrightarrow{[O]} RSSR}$

(a) R = alkyl or aryl group; Ar = aryl group; Y = oxygen or sulfur.

TABLE B-6. POSSIBLE CONTAMINANTS IN AZINPHOSMETHYL

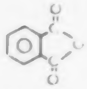
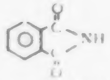
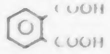
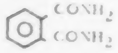
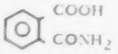
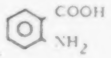
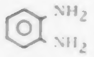
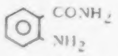
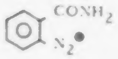
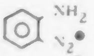
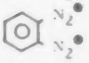
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	P_2S_5	SM	–	1	o
2	$(CH_3O)_2P(S)SH$	1	1	1	+
3	$(CH_3O)_2(CH_3S)PS$	2	2	1	++
4	$(CH_3O)_3PS$	2 3 8	3 4 8	1 1 1	++
5	$(CH_3O)(CH_3S)_2PS$	3	5	1	++
6	$(CH_3O)(CH_3S)P(S)SH$	2	5	1	+
7	$(CH_3O)_2P(O)OH$	9	6	1 or St	+
8	$(CH_3O)_2P(S)OH$	2 70	6 35	1 St	+
9	$(CH_3O)_2P(O)SH$	8	7	1	+
10	$(CH_3O)_2P(S)SP(S)(OCH_3)_2$	2	9	1	++
11	$(CH_3O)_2P(S)SSP(S)(OCH_3)_2$	2	10	1	+
12	$(CH_3O)_2PSH$	2	11	1	o
13	$(CH_3O)_3P$	12	12	1	+
14	$(CH_3O)_2(CH_3S)P$	12	13	1	+
15	$(CH_3O)_2P(S)SP(OCH_3)_2$	12	14	1	o
16	$(CH_3O)_2(CH_3S)PO$	9	2	1	+
17	$(CH_3O)_3PO$	9	3	1	+
18	$(CH_3O)(CH_3S)P(S)SH$	2	5	1	o
19		SM	–	2	o
20		19	18	2	o
21		19	19	2	+
22		20 23	20 23	2 4	+
23		20 21	21 23	2 4	+
24		23	22	3	+
25		22	22	3	o
26		24	23	4	++
27		26	24	5	o
28		25	24	5	o
29		28	24	5	o

TABLE B-6. (Continued)

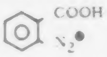
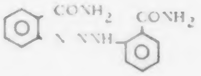
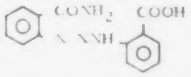
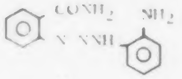
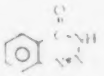
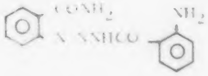
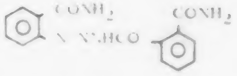
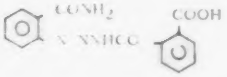
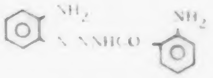
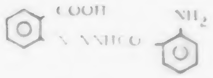
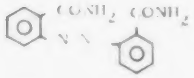
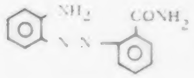
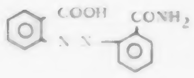
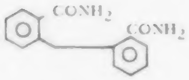
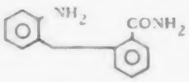
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
30		24	24	5	o
31		27 + 26	25	5	+
32		27 + 24	25	5	+
33		27 + 25	25	5	o
34		27	26	5	++
35		27 + 26	26	5	+
36		27 + 22	26	5	o
37		27 + 23	26	5	+
38		28 + 26	26	5	o
39		30 + 26	26	5	+
40		27	27	5	+
41		28 + 27	27	5	o
42		30 + 27	27	5	o
43		27	28	5	+
44		28 + 27	28	5	o

TABLE B-6. (Continued)

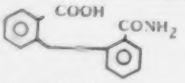
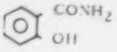
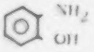
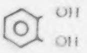
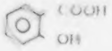
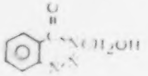
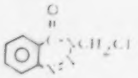
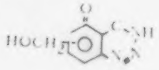
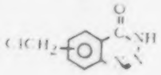
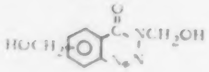
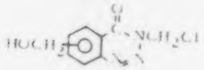
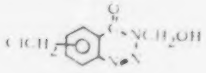
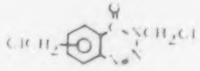
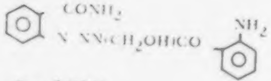
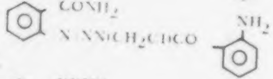
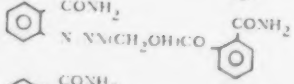
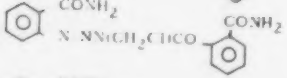
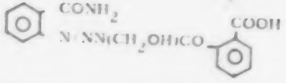
No.	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
45		30 + 27	28	5	o
46		27	29	5	+
47		28	29	5	o
48		29	29	5	o
49		30	29	5	o
50	$(CH_2O)_x$	SM	—	6	++
51	$ClCH_2OCH_2Cl$	50	30	6	+
52		34 71	31 35	6 St	+
53		52	33	6	++
54		34	32	6	o
55		54	33	6	+
56		52	32	6	o
57		53 56	32 33	6 6	o
58		56 55	33 32	6 6	o
59		58 57	32 32	6 6	+
60		35	31	6	o
61		60	33	6	o
62		36	31	6	o
63		62	33	6	o
64		37	31	6	o

TABLE B- 6. (Continued)

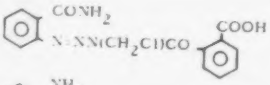
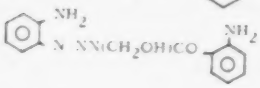
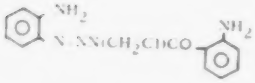
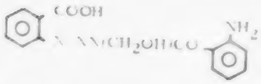
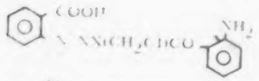
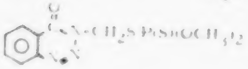
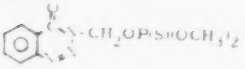
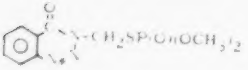
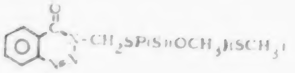
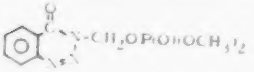
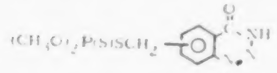
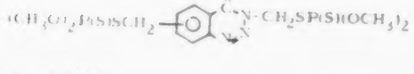
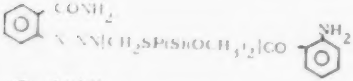
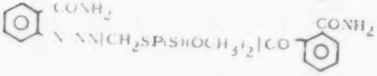
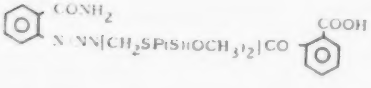
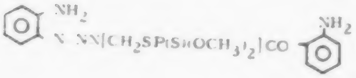
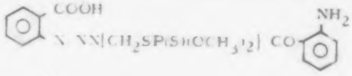
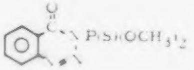
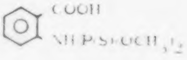
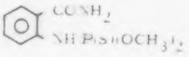
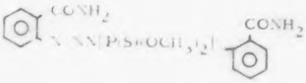
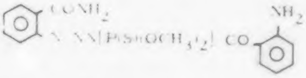
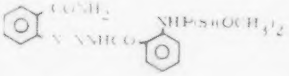
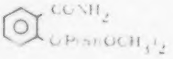
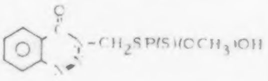
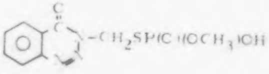
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
65		64	33	6	o
66		38	31	6	o
67		66	33	6	o
68		39	31	6	o
69		68	33	6	o
70		52 + 2 53 + 2	2 15	6 6	Azinphos-methyl
71		52 + 8 52 - 2 72	8 3 36	6 6 St	++
72		52 + 9 53 + 9 70	2 15 34	6 6 St	++
73		52 + 6 53 + 6	2 15	6 6	++
74		52 + 7 52 + 9	8 3	6 6	+
75		54 + 2 55 + 2	2 15	6 6	+
76		56 + 2 57 + 2 58 + 2 59 + 2	2 2 + 15 2 + 15 15	6 6 6 6	+
77		60 + 2 61 + 2	2 15	6 6	o
78		62 + 2 63 + 2	2 15	6 6	o
79		64 65	2 15	6 6	o
80		66 67	2 15	6 6	o

TABLE B-6. (Continued)

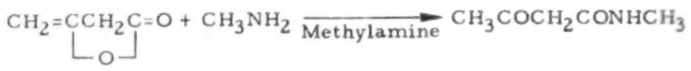
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
81		68 69	2 15	6 6	0
82		34 + 2	17	6	+
83		24 + 2	17	6	+
84		26 + 2	17	6	++
85		31 + 2	17	6	0
86		35 + 2	17	6	0
87		35 + 2	17	6	+
88		46 + 2	3	6	+
89	$(CH_3O)_2P(S)SCH_2OCH_2Cl$	51 + 2	15	6	+
90	$(CH_3O)_2P(S)SCH_2OCH_2SP(S)(OCH_3)_2$	51 + 2	15	6	+
91		70	35	St	+
92		91	7 + 6	St	+
93	CH_3SH	73	35	St	+
94	CH_3SSCH_3	93	37	St	+

(a) Precursor refers to the contaminant numbered in this table. SM = starting material.

(b) See Table B-5.

(c) The step (Figure B-3) during which the contaminant might be formed. St indicates possible formulation during storage.

(d) 0 = likely present only at levels below 1 ppm
+ = likely present at levels above 1 ppm but below 100 ppm
++ = likely present at levels above 100 ppm.

Step 1. 

$CH_2{=}\underbrace{CCH_2C}_{O}{=}O + CH_3NH_2 \xrightarrow[\text{Methylamine}]{} CH_3COCH_2CONHCH_3$

Ketene Dimer N-Methylacetoacetamide

Step 2. $CH_3COCH_2CONHCH_3 + SO_2Cl_2 \rightarrow CH_3COCHClCONHCH_3 + SO_2 + HCl$

Sulfuryl Chloride 2-Chloro-N-Methylacetacetamide

Step 3. $PCl_3 + CH_3OH \rightarrow (CH_3O)_3P$

Trimethyl Phosphite

Step 4. $CH_3COCHClCONHCH_3 + (CH_3O)_3P \rightarrow (CH_3O)_2P(O)CH(CH_3){=}CHCONHCH_3 + CH_3Cl$

AZODRIN

FIGURE B-4. SYNTHESIS OF AZODRIN

TABLE ~~B~~-7. REACTIONS OCCURRING DURING PRODUCTION AND STORAGE OF AZODRIN

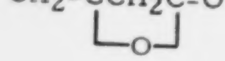
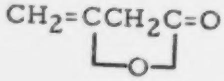
Reaction Number	Generalized Reaction
1.	 $CH_2=CCH_2C=O$ (ring: –O–) $+ RNH_2 \rightarrow CH_3COCH_2CONHR$
2.	 $CH_2=CCH_2C=O$ (ring: –O–) $+ H_2O \rightarrow CH_3COCH_2COOH$
3.	$CH_3COCH_2COOH \rightarrow CH_3COCH_3 + CO_2$
4.	$RCH_2COR' + SO_2Cl_2 \rightarrow RCHClCOR' + SO_2 + HCl$
5.	$RCHClCOR + SO_2Cl_2 \rightarrow RCCl_2COR' + SO_2 + HCl$
6.	$ROH + PCl_3 \rightarrow ROPCl_2 + HCl$
7.	$ROPCl_2 + ROH \rightarrow (RO)_2PCl + HCl$
8.	$(RO)_2PCl + ROH \rightarrow (RO)_3P + HCl$
9.	$(RO)_3P + HCl \rightarrow (RO)_2POH + RCl$
10.	$(RO)_2POH + HCl \rightarrow ROP(OH)_2 + RCl$
11.	$(RO)_3P \xrightarrow{[O]} (RO)_3PO$
12.	$(RO)_3P + R'CH_2COCHClR' \rightarrow (RO)_2P(O)OC(R'CH_2)=CHR' + RCl$
13.	$(RO)_3PO + H_2O \rightarrow (RO)_2P(O)OH + ROH$
14.	$(RO)_2P(O)OH + H_2O \rightarrow ROP(O)(OH)_2 + ROH$
15.	$(RO)_2P(O)OC(R'CH_2)=CHR'' \xrightarrow{\Delta} (RO)_2P(O)OH + [R'CH=C=CHR'']_x$
16.	$RCONHR' + H_2O \rightarrow RCOOH + R'NH_2$
17.	$(RO)_3PO + R'NH_2 \rightarrow (RO)_2P(C)NHR' + ROH$

TABLE B-8. POSSIBLE CONTAMINANTS IN AZODRIN

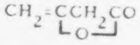
Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	 $\underset{\llcorner O \lrcorner}{CH_2{=}CCH_2CO}$	SM	–	1	0
2	CH_3NH_2	SM	–	1	0
3	$CH_3COCH_2CONHCH_3$	1+2	1	1	0
4	CH_3COCH_2COOH	1	2	1	0
5	CH_3COCH_3	4	3	1	0
6	SO_2Cl_2	SM	–	2	0
7	$CH_3COCHClCONHCH_3$	3	4	2	++
8	$CH_3COCCl_2CONHCH_3$	7	5	2	+
9	$ClCH_2COCH_2CONHCH_3$	3	4	2	+
10	$ClCH_2COCHClCONHCH_3$	7	4	2	0
11	CH_3COCH_2Cl	5	4	2	0
12	$ClCH_2COCH_2Cl$	11	4	2	0
13	PCl_3	SM	–	3	0
14	CH_3OH	SM	–	3	0
15	$(CH_3O)PCl_2$	13+14	6	3	0
16	$(CH_3O)_2PCl$	14+15	7	3	0
17	$(CH_3O)_3P$	14+16	8	3	+
18	$(CH_3O)_2P(O)H$	17	9	3	+
19	$CH_3OP(OH)_2$	18	10	3	+

TABLE B-8. (Continued)

Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
20	$P(OH)_3$	19	10	3	0
21	CH_3Cl	17+7	12	4	0
22	$(CH_3O)_3PO$	17	11	3 or St	+
23	$(CH_3O)_2P(O)OC(CH_3)=CHCONHCH_3$	17+7	12	4	Azodrin
24	$(CH_3O)_2P(O)OC(CH_3)=CClCONHCH_3$	17+8	12	4	+
25	$(CH_3O)_2P(O)OC(CH_2CONHCH_3)=CH_2$	17+9	12	4	+
26	$(CH_3O)_2P(O)OC(CH_2Cl)=CHCONHCH_3$	17+10	12	4	0
27	$(CH_3O)_2P(O)OC(CH_3)=CH_2$	17+11	12	4	+
28	$(CH_3O)_2P(O)OC(CH_2Cl)=CH_2$	17+12	12	4	0
29	$(CH_3O)_2P(O)OH$	23	13	4 or St	++
30	$CH_3OP(O)(OH)_2$	29	13	4 or St	++
31	$[CH_2=C-CONHCH_3]_x$	23	15	4 or St	++
32	$(CH_3O)_2P(O)OC(CH_3)=CHCOOH$	23	16	4 or St	+
33	$(CH_3O)_2P(O)NHCH_3$	23+2	17	4 or St	+
34	$(CH_3O)(CH_3NH)P(O)OC(CH_3)=CHCONHCH_3$	23+2	17	4 or St	+

(a) Precursor refers to the contaminant numbered in this table
SM = starting material.

(b) See Table B-7.

(c) The step (Figure B-4) during which the contaminant might be formed.
St indicates possible formation during storage.

(d) 0 = likely to be present below 1 ppm.
+ = likely to be present above 1 ppm but below 100 ppm.
++ = likely to be present at levels above 100 ppm.

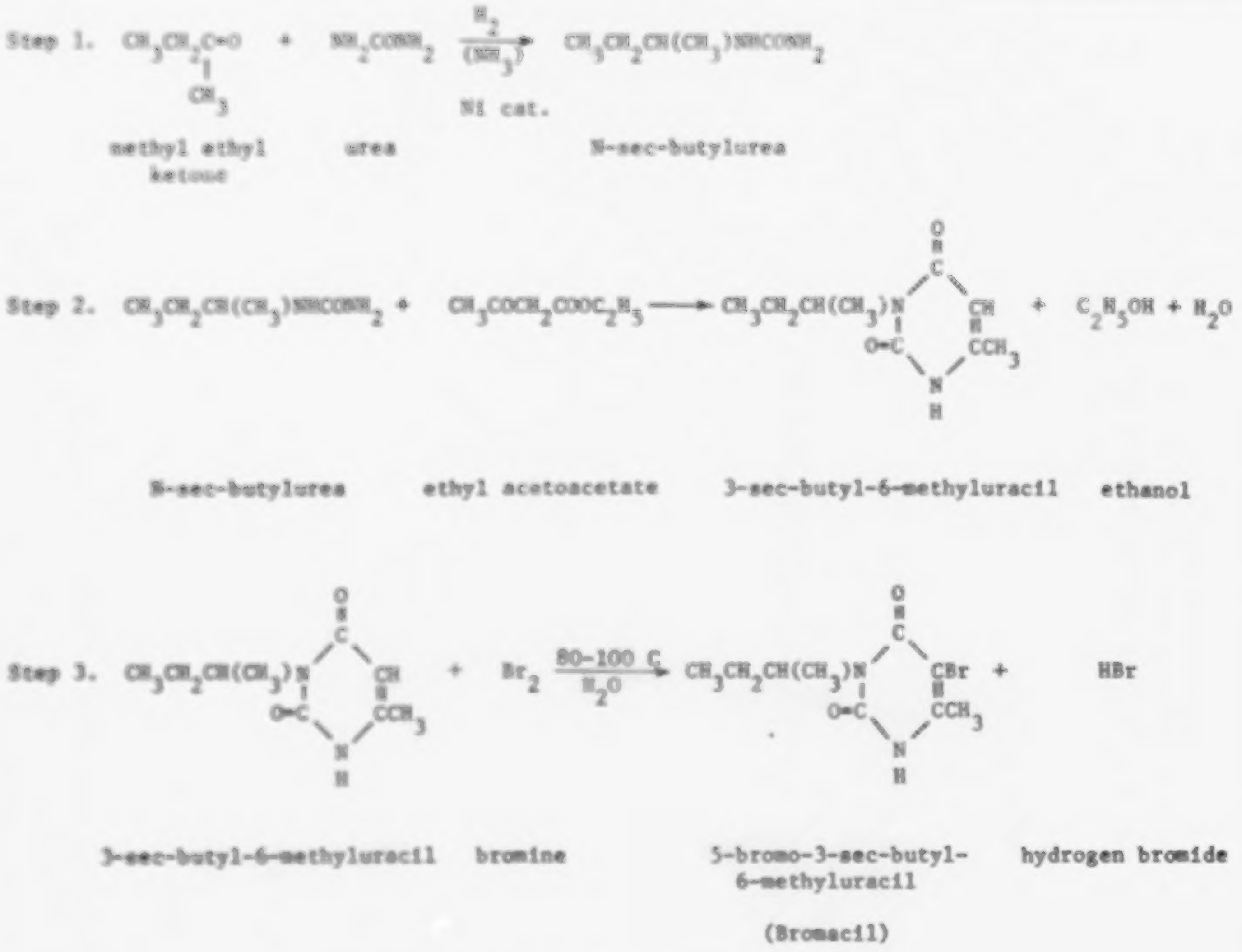

Step 1.
methyl ethyl ketone
urea
Ni cat.
N-sec-butylurea
Step 2.
N-sec-butylurea
ethyl acetoacetate
3-sec-butyl-6-methyluracil
ethanol
Step 3.
3-sec-butyl-6-methyluracil
bromine
80-100 C
5-bromo-3-sec-butyl-6-methyluracil
(Bromacil)
hydrogen bromide

FIGURE B-5. SYNTHESIS OF BROMACIL

TABLE B-9. REACTIONS OCCURRING DURING PRODUCTION OF BROMACIL

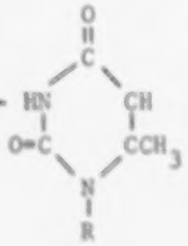
Reaction Number	Generalized Reaction
1	$RR'CO + R''NHCONH_2 + H_2 \xrightarrow[NH_3]{Ni\ cat.} RR'CHNHCONHR'' + H_2O$
2	$RR'CO + H_2 \xrightarrow{Ni\ cat.} RR'CHOH$
3	$RR'CO + R''NH_2 + H_2 \xrightarrow{Ni\ cat.} RR'CHNHR'' + H_2O$
4	$RNHCONHR' + CH_3COCH_2COOR'' \longrightarrow RNHCONR'C(CH_3){=}CHCOOR'' + H_2O$
5	$RNHCONR'C(CH_3){=}CHCOOR'' \longrightarrow$  $+ R''OH$
6	$CH_3COCH_2COOR + H_2O \longrightarrow CH_3COCH_3 + CO_2 + ROH$
7	$RH + Br_2 \longrightarrow RBr + HBr$
8	$RBr + R'NH_2 \longrightarrow RNHR' + HBr$
9	$RCOOR' + R''NH_2 \longrightarrow RCONHR'' + R'OH$
10	$RBr + H_2O \longrightarrow ROH + HBr$

TABLE B-10. POSSIBLE CONTAMINANTS IN BROMACIL

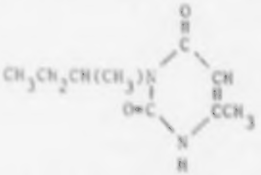
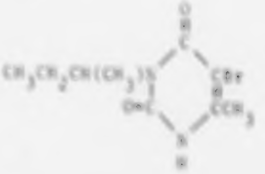
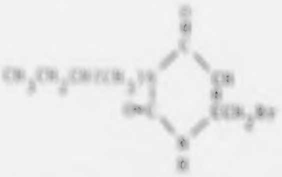
Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	NH_2CONH_2	SM	--	1	+
2	$CH_3CH_2COCH_3$	SM	--	1	0
3	NH_3	cat.	--	1	0
4	$CH_3CH_2CH(CH_3)NHCONH_2$	1 + 2	1	1	++
5	$CH_3CH_2CH(CH_3)NHCONHCH(CH_3)CH_2CH_3$	2 + 4	1	1	++
6	$CH_3CH_2CH(CH_3)OH$	2	2	1	0
7	$CH_3CH_2CH(CH_3)NH_2$	2 + 3	3	1	0
8	$CH_3CH_2CH(CH_3)NHCH(CH_3)CH_2CH_3$	2 + 7	3	1	0
9	$CH_3COCH_2COOC_2H_5$	SM	--	2	+
10	$CH_3CH_2CH(CH_3)NHCONHC(CH_3){=}CHCOOC_2H_5$	4 + 9	4	2	+
11		10	5	2	++
12	CH_3COCH_3	9	6	2	0
13	C_2H_5OH	10	5	2	0
14	Br_2	SM	--	3	0
15		11 + 14	7	3	Bromacil
16	HBr	11 + 14	7	3	0
17		11 + 14	7	3	+

TABLE B-10. (Continued)

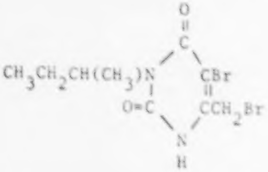
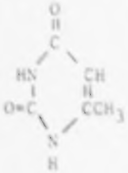
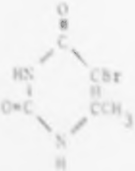
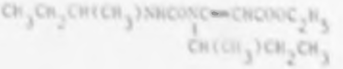
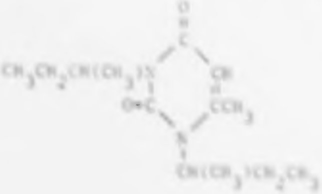
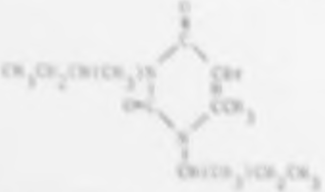
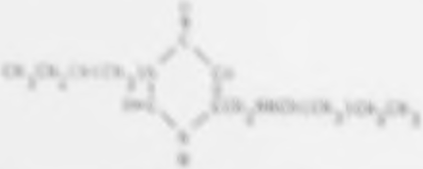
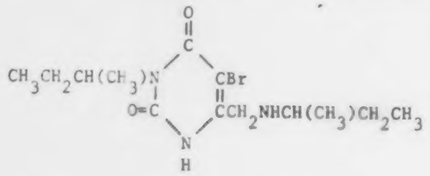
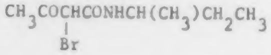
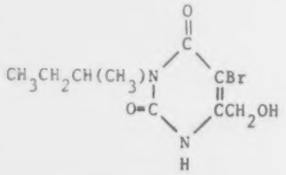
Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
18		14 + 15 or 14 + 17	7	3	+
19	$H_2NCONHC(CH_3){=}CHCOOC_2H_5$	1 + 9	4	2	+
20		19	5	2	+
21		20	7	3	+
22	 $CH_3CH_2CH(CH_3)NHCON\underset{CH(CH_3)CH_2CH_3}{\underset{\vert}{C}}{=}CHCOOC_2H_5$	5 + 9	4	2	+
23		22	5	2	+
24		23	7	3	+
25	$CH_3COCHBrCOOC_2H_5$	9 + 14	7	3	0
26		13 + 7	8	3	0

TABLE B-10. (Continued)

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
27		18 + 7	8	3	+
28	$CH_3COCH[CH(CH_3)CH_2CH_3]COOC_2H_5$	25 + 7	8	3	0
29	$CH_3COCH_2CONHCH(CH_3)CH_2CH_3$	9 + 7	9	2	0
30	 $CH_3CO\underset{\mid\atop Br}{C}HCONHCH(CH_3)CH_2CH_3$	29 + 14	7	3	0
31	$CH_3COCH[CH(CH_3)CH_2CH_3]CONHCH(CH_3)CH_2CH_3$	30 + 7	8	3	0
32		18	10	3	+

(a) Precursor refers to the contaminant numbered in this table. SM = starting material; cat. = catalyst.
(b) See Table B-9.
(c) The step (Figure B-5) during which the contaminant might be formed.
(d) 0 = likely to be present at levels below 1 ppm
+ = likely to be present at levels above 1 ppm but below 100 ppm
++ = likely to be present at levels above 100 ppm.

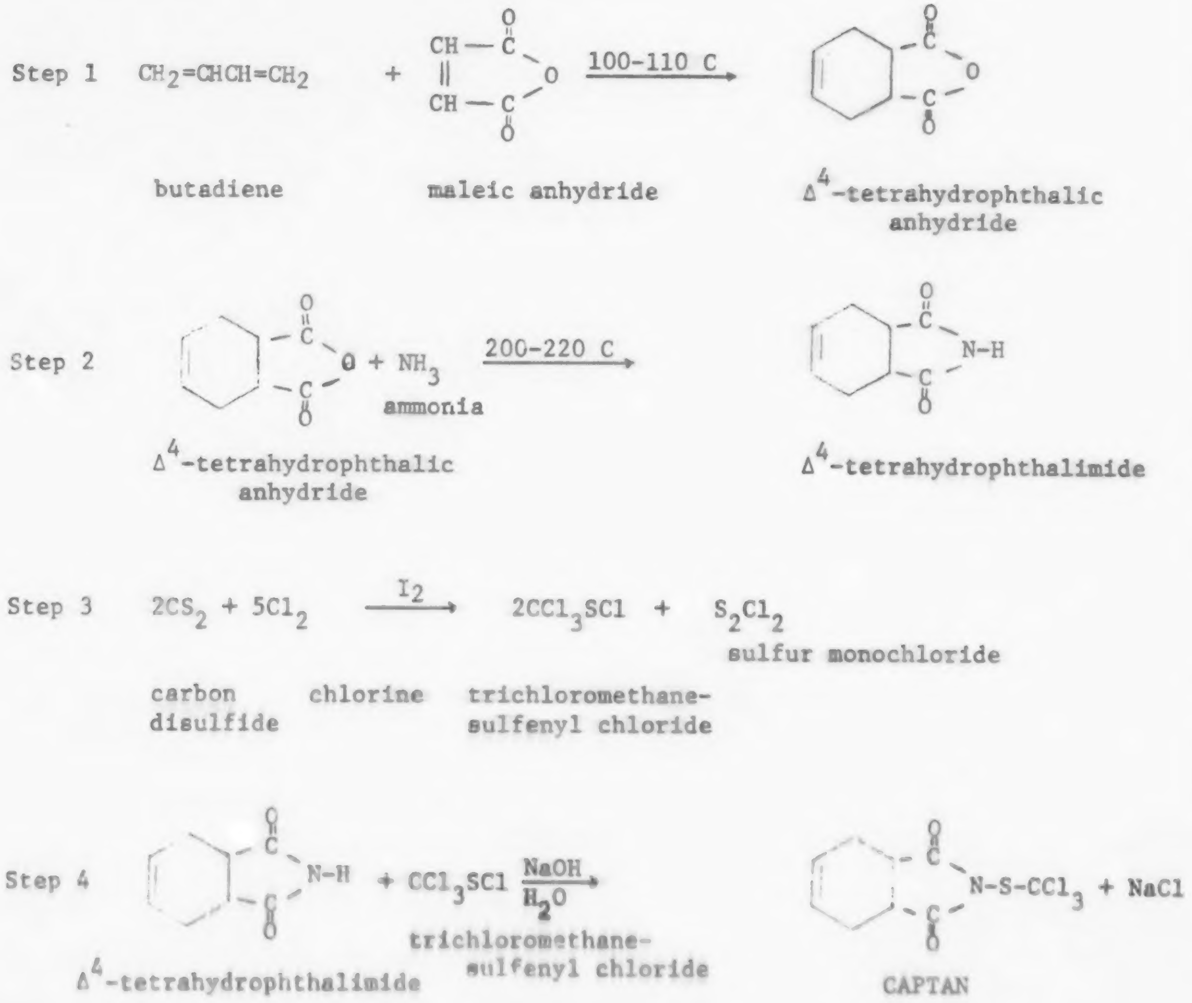
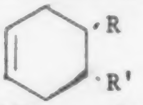

Step 1
CH2=CHCH=CH2
butadiene
maleic anhydride
100-110 C
Δ4-tetrahydrophthalic anhydride
Step 2
Δ4-tetrahydrophthalic anhydride
+ NH3
ammonia
200-220 C
Δ4-tetrahydrophthalimide
Step 3
2CS2 + 5Cl2
I2
2CCl3SCl + S2Cl2
sulfur monochloride
carbon disulfide
chlorine
trichloromethane-sulfenyl chloride
Step 4
Δ4-tetrahydrophthalimide
+ CCl3SCl
NaOH
H2O
trichloromethane-sulfenyl chloride
N-S-CCl3 + NaCl
CAPTAN

FIGURE B-6. MAIN REACTIONS IN THE PREPARATION OF CAPTAN

TABLE B-11. REACTIONS OCCURRING DURING PRODUCTION OF CAPTAN

Reaction Number	Generalized Reaction[a]
1	$CH_2=CH-CH=CH_2 + RCH=CHR' \longrightarrow$ 
2	$(RCO)_2O + NH_3 \longrightarrow RCONH_2 + RCOOH \rightleftharpoons (RCO)_2NH + H_2O$
3	$(RCO)_2O + H_2O \longrightarrow 2\ RCOOH$
4	$(RCO)_2NH + NH_3 \longrightarrow 2\ RCONH_2$
5	$RCOOH + NH_3 \xrightarrow{\Delta} RCONH_2 + H_2O$
6	$2CS_2 + 3Cl_2 \xrightarrow{I2} 2CSCl_2 + S_2Cl_2$
7	$CSCl_2 + Cl_2 \longrightarrow CCl_3SCl$
8	$CSCl_2 + 3Cl_2 + 2H_2O \longrightarrow CCl_3SO_2Cl + 4HCl$
9	$2S_2Cl_2 + 2H_2O \longrightarrow 3S + 4HCl + SO_2$
10	$4CCl_3SCl \xrightarrow{Fe} S_2Cl_2 + 2CCl_4 + CCl_3SSCCl_3$
11	$2CCl_3SCl + Cl_2 \longrightarrow 2CCl_4 + S_2Cl_2$
12	$CCl_3SCl + H_2O \longrightarrow CCl_3SOH + HCl$
13	$CCl_3SOH + H_2O \longrightarrow CO_2 + S + 3HCl$
14	$3CCl_3SOH \longrightarrow CCl_3SSCCl_3 + CCl_3SO_2H + H_2O$
15	$CCl_3SOH \xrightarrow{[0]} CCl_3SO_2H$
16	$CCl_3SCl + (RCO)_2NH \xrightarrow{NaOH} (RCO)_2NSCCl_3$
17	$CCl_3SO_2Cl + (RCO)_2NH \xrightarrow{NaOH} (RCO)_2NSO_2CCl_3$
18	$CCl_3SCl + RCH=CHR \longrightarrow R(CCl_3S)CHCHClR$
19	$RCH=CHR + CCl_3SO_2Cl \longrightarrow RCHClCHRCCl_3 + SO_2$
20	$CSCl_2 + (RCO)_2NH \longrightarrow (RCO)_2NCSN(COR)_2$
21	$(RCO)_2NSCCl_3 + H_2O \longrightarrow (RCO)_2NH + CCl_3SOH$
22	$(RCO)_2NSCCl_3 \xrightarrow{[0]} (RCO)_2NSO_2CCl_3$
23	$(RCO)_2NH + S_2Cl_2 \longrightarrow (RCO)_2NSSN(COR)_2$
24	$(RCO)_2NH + 2CCl_3SCl \qquad (RCO)_2NCl + CCl_3SSCCl_3 + HCl$
25	$RCONH_2 + 2CCl_3SCl \qquad RCONHCl + CCl_3SSCCl_3 + HCl$

a. R = alkyl or aryl group

TABLE B-12. POSSIBLE CONTAMINANTS IN CAPTAN

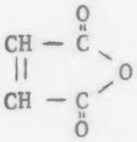
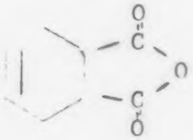
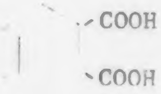
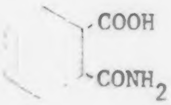
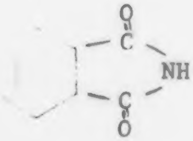
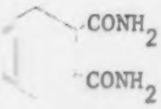
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1.	$CH_2=CHCH=CH_2$	SM	--	1	0
2.		SM	--	1	0
3.		1+2	1	1	+
4.	HOOCCH=CHCOOH	2	3	1	0
5.		3 1+4	3 1	1 1	+
6.		3 5	2 5	2 2	+
7.		6 22	2 21	2 St	++
8.		7 6	4 5	2 2	+
9.	$HOCCH=CHCONH_2$	2 4	2 5	2 2	0

TABLE B-12. (Continued)

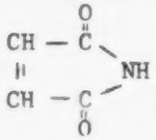
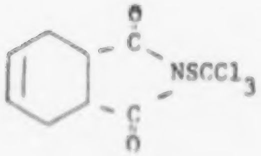
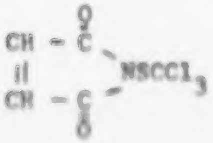
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
10.		9	2	2	0
11.	$H_2NOCCH=CHCONH_2$	10 9	4 5	2 2	0
12.	CS_2	SM	--	3	0
13.	$CSCl_2$	12	6	3	0
14.	S_2Cl_2	12 15	6 10	3 3	0
15.	CCl_3SCl	13	7	3	+
16.	S	14 20	9 13	3 4 or St	++
17.	CCl_4	15 15	10 11	3 3	0
18.	CCl_3SSCCl_3	15 20	10 14	3 4 or St	+
19.	CCl_3SO_2Cl	12	8	3	0
20.	CCl_3SOH	15 22	12 21	4 4 or St	0
21.	CCl_3SO_2H	20 20	15 14	4 or St 4 or St	+
22.		7+15	16	4	CAPTAN
23.		10+15	16	4	+

TABLE B-12. (Continued)

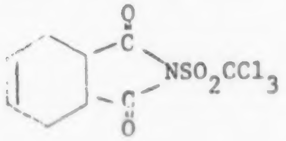
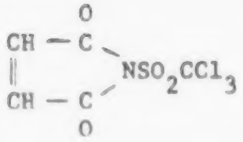
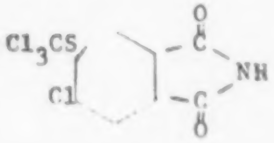
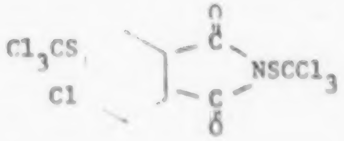
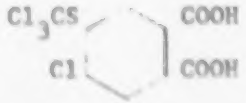
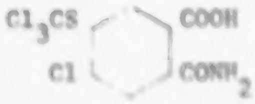
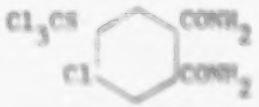
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
24.		7+19 22	17 22	4 4 or st	++
25.		10+19 23	17 22	4 4 or st	0
26.		7+15	18	4	0
27.		22+15 26+15	18 16	4 4	+
28.		5+15	18	4	0
29.		6+15	18	4	0
30.		8+15	18	4	0

TABLE B-12. (Continued)

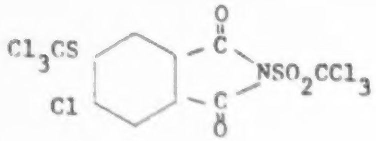
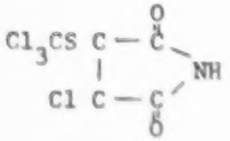
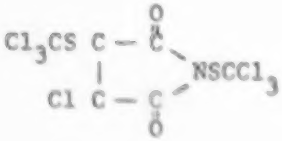
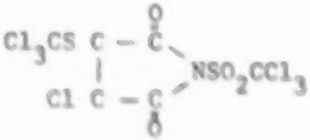
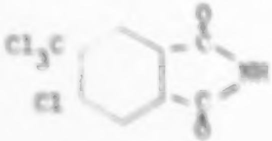
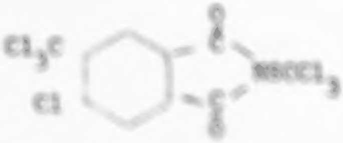
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
31.	 Cl_3CS Cl O C NSO_2CCl_3 C O	24+15 26+19	18 17	4 4	0
32.	 Cl_3CS C Cl C O C NH C O	10+15	18	4	0
33.	 Cl_3CS C Cl C O C $NSCCl_3$ C O	23+15 32+15	18 16	4 4	0
34.	 Cl_3CS C Cl C O C NSO_2CCl_3 C O	25+15 32+19	18 17	4 4	0
35.	HOOC $CH(SCCl_3)CHClCOOH$	4+15	18	4	0
36.	HOOC $CH(SCCl_3)CHClCONH_2$	9+15	18	4	0
37.	$H_2NOCCH(SCCl_3)CHClCONH_2$	11+15	18	4	0
38.	 Cl_3C Cl O C NH C O	7+19	19	4	0
39.	 Cl_3C Cl O C $NSCCl_3$ C O	22+19 38+15	19 16	4 4	0

TABLE B-12. (Continued)

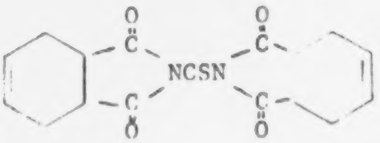
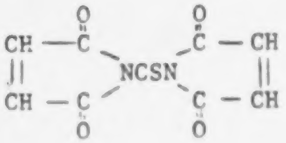
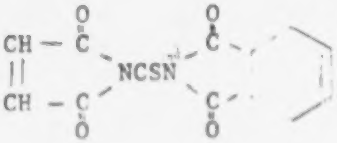
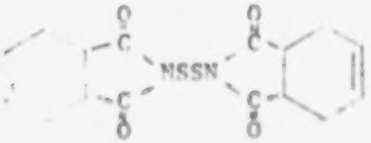
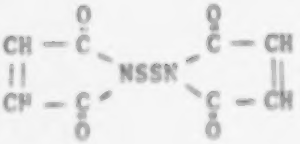
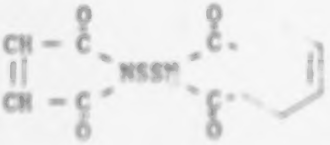
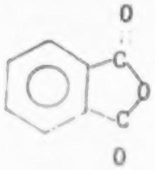
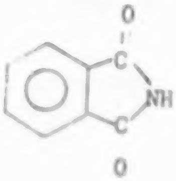
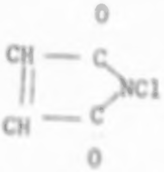
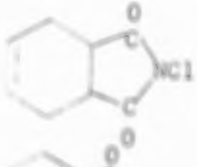
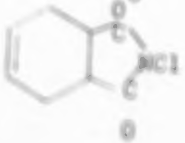
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
40.		7+13	20	4	+
41.		10+13	20	4	0
42.		7+10+13	20	4	0
43.		7+14	23	4	+
44.		10+14	23	4	0
45.		7+10+14	23	4	0

TABLE B-12. (Continued)

No.	Contaminant	Precursor[a]	Reaction Number[b]	Step [c]	Likelihood of Being Present in Final Product[d]
46	$HOOCCOOH$	2 (Impurity)	-	1	0
47		2 (Impurity)	-	1	+
48	$HOOCCONH_2$	46	5	2	+
49		47	2	2	+
50	$HOOCCONHCl$	48	25	4	+
51	$HOOCCH = CHCONHCl$	9	25	4	+
52		10	24	4	+
53		7	24	4	+
54		49	24	4	+

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a. Precursor refers to the contaminant numbered in this table. SM = starting material

b. See Table B-11.

c. The step (Figure B-6) during which the contaminant might be formed. St indicates possible formation during storage.

d. 0 = likely present only at levels below 1 ppm.
  + = likely present at levels above 1 ppm but below 100 ppm.
 ++ = likely present at levels above 100 ppm.

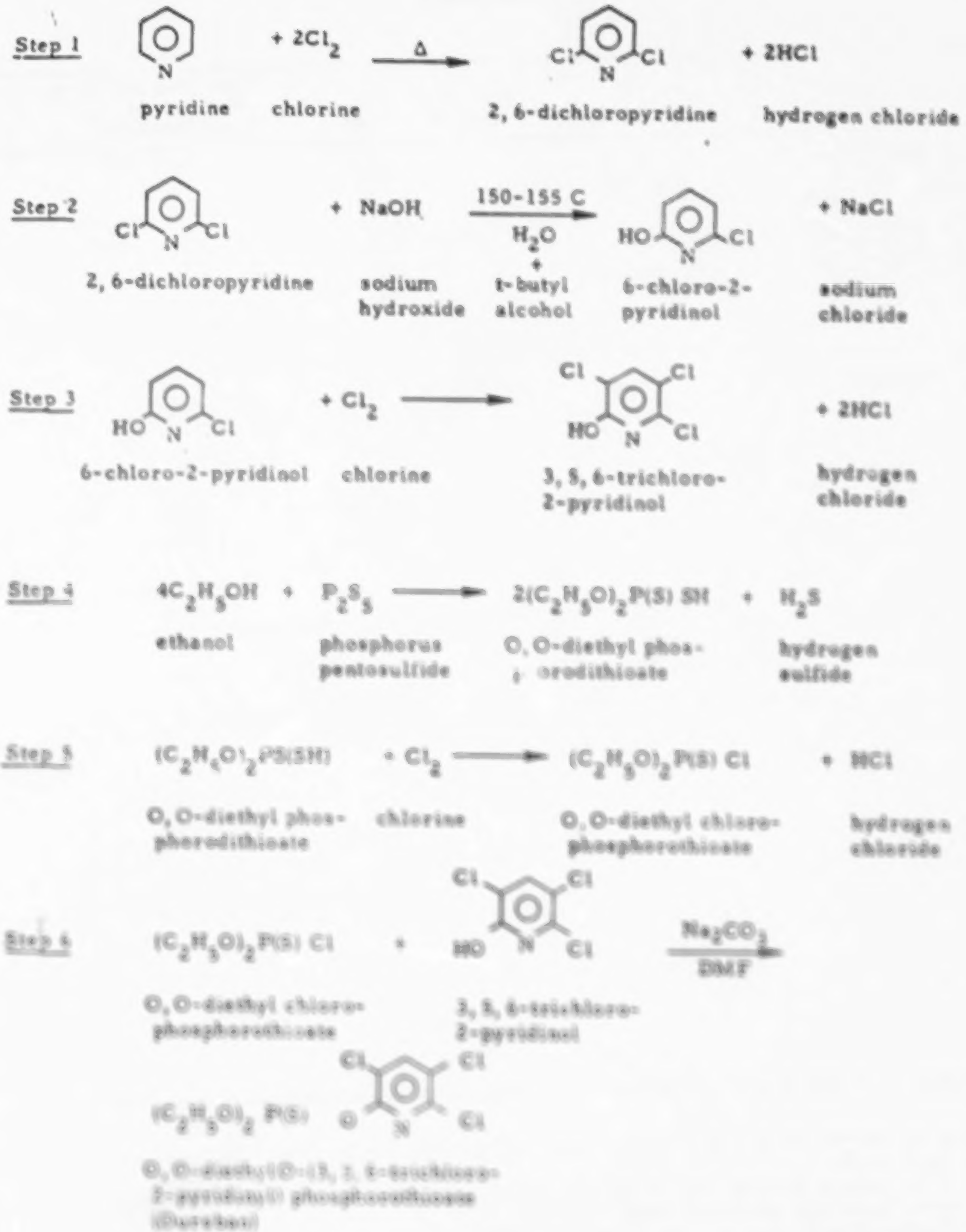
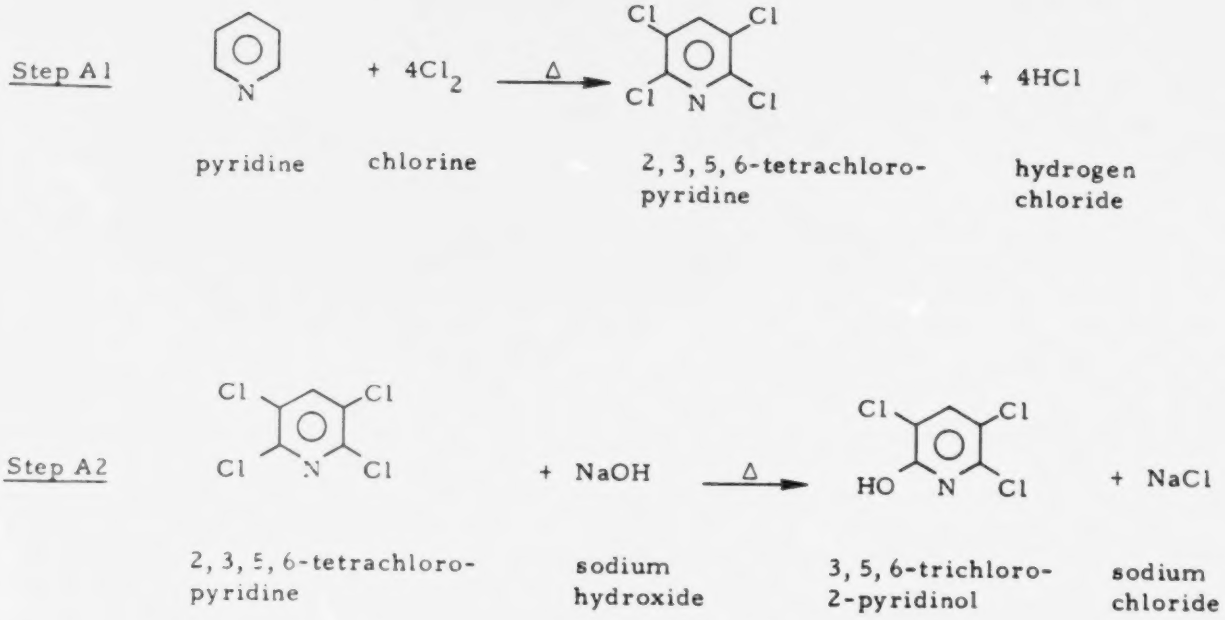

Step 1
+ 2Cl2
Δ
+ 2HCl
pyridine
chlorine
2, 6-dichloropyridine
hydrogen chloride
Step 2
+ NaOH
150-155 C
H2O + t-butyl alcohol
+ NaCl
2, 6-dichloropyridine
sodium hydroxide
6-chloro-2-pyridinol
sodium chloride
Step 3
+ Cl2
+ 2HCl
6-chloro-2-pyridinol
chlorine
3, 5, 6-trichloro-2-pyridinol
hydrogen chloride
Step 4
4C2H5OH + P2S5
2(C2H5O)2P(S) SH + H2S
ethanol
phosphorus pentosulfide
O, O-diethyl phosphorodithioate
hydrogen sulfide
Step 5
(C2H5O)2PS(SH) + Cl2
(C2H5O)2P(S) Cl + HCl
O, O-diethyl phosphorodithioate
chlorine
O, O-diethyl chlorophosphorothioate
hydrogen chloride
Step 6
(C2H5O)2P(S) Cl +
Na2CO3
DMF
O, O-diethyl chlorophosphorothioate
3, 5, 6-trichloro-2-pyridinol
(C2H5O)2 P(S)
O, O-diethyl O-(3, 5, 6-trichloro-2-pyridinyl) phosphorothioate
(Dursban)

FIGURE 8-7. SYNTHESIS OF CHLORPYRIFOS



Step A1
N
+ 4Cl2
Δ
Cl
Cl
Cl
N
Cl
+ 4HCl
pyridine
chlorine
2, 3, 5, 6-tetrachloro-pyridine
hydrogen chloride
Step A2
Cl
Cl
Cl
N
Cl
+ NaOH
Δ
Cl
Cl
HO
N
Cl
+ NaCl
2, 3, 5, 6-tetrachloro-pyridine
sodium hydroxide
3, 5, 6-trichloro-2-pyridinol
sodium chloride

FIGURE B-8. ALTERNATE SYNTHESIS OF 3,5,6-TRICHLORO-2-PYRIDINOL

TABLE B-13. REACTIONS OCCURRING DURING PRODUCTION OF CHLORPYRIFOS

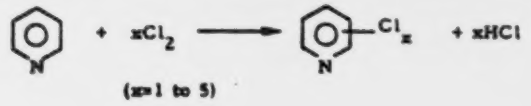
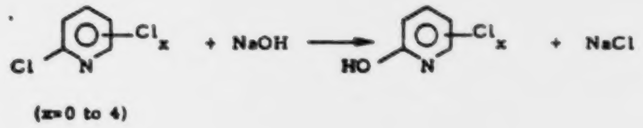
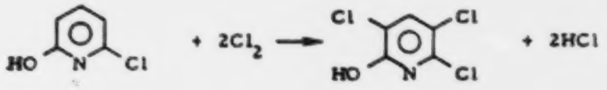
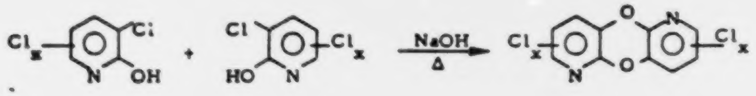
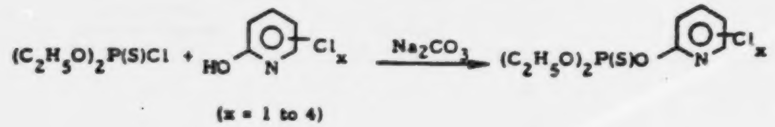
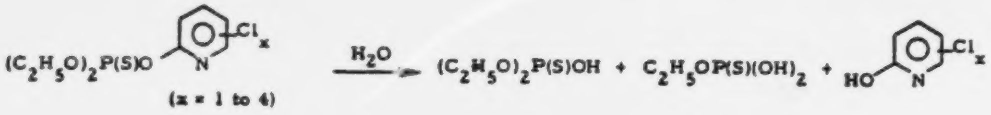
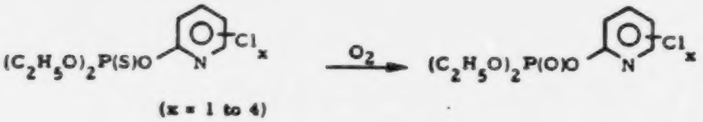
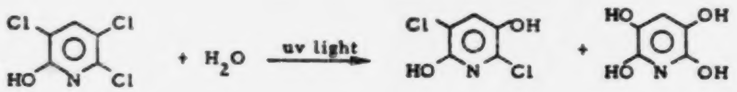
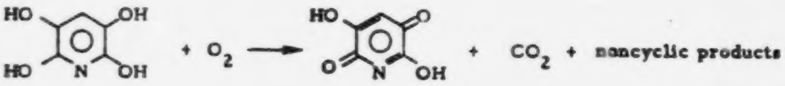
Reaction Number	Generalized Reaction[a]
1	 + xCl2 (x=1 to 5) N Cl x + xHCl
2	 Cl N Cl x + NaOH HO (x=0 to 4) + NaCl
3	 HO N Cl + 2Cl2 + 2HCl
4	 Cl x Cl N OH + HO NaOH Δ O
5	$4\,C_2H_5OH + P_2S_5 \rightarrow 2(C_2H_5O)_2P(S)SH + H_2S$
6	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)SR' + H_2O$
7	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)OR' + H_2S$
8	$(RY)_2P(Y)SR + R'OH \rightarrow (RY)_2P(Y)OR' + RSH$
9	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
10	$(RY)_2P(Y)SH + H_2O \rightarrow (RY)_2P(Y)OH + H_2S$
11	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
12	$2(RY)_2P(Y)YH \rightarrow (RY)_2P(Y)YP(Y)(YR)_2 + H_2Y$
13	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(YR)_2$
14	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
15	$(RY)_2P(S)SH \rightarrow (RY)_2PSH + S$
16	$(RY)_2PSH + R'OH \rightarrow (RY)_2POR' + H_2S$
17	$(RY)_2PSH + R'OH \rightarrow (RY)_2PSR' + H_2O$
18	$2(RY)_2P(S)SH + Cl_2 \rightarrow (RY)_2P(S)SSP(S)(YR)_2 + 2HCl$
19	$(RY)_2P(S)SH + Cl_2 \rightarrow (RY)_2P(S)Cl + S_2Cl_2$

TABLE B-13. (Continued)

Reaction Number	Generalized Reaction[(a)]
20	 (C2H5O)2P(S)Cl HO N Clx Na2CO3 (C2H5O)2P(S)O (x = 1 to 4)
21	 (C2H5O)2P(S)O N Clx (x = 1 to 4) H2O (C2H5O)2P(S)OH + C2H5OP(S)(OH)2 + HO
22	 (C2H5O)2P(S)O N Clx (x = 1 to 4) O2 (C2H5O)2P(O)O
23	 Cl HO N + H2O uv light OH +
24	 HO OH N + O2 O + CO2 + noncyclic products

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(a) R and R' = ethyl or substituted pyridyl group
Y = oxygen or sulfur.

TABLE B-14. POSSIBLE CONTAMINANTS IN CHLORPYRIFOS

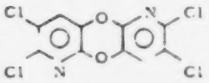
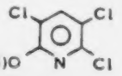
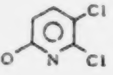
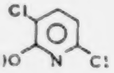
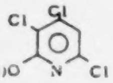
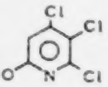
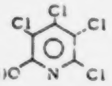
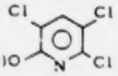
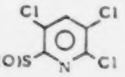
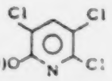
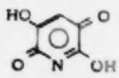
Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
1	C_5H_5N	SM	–	1	o
2	Cl_2	SM	–	1	o
3	2-Cl C_5H_4N	1+2	1	1	o
4	2,6-Cl_2 C_5H_3N	1+2	1	1	o
5	2,3,6-Cl_3 C_5H_2N	1+2	1	1	o
6	2,4,6-Cl_3 C_5H_2N	1+2	1	1	o
7	2,3,4,6-Cl_4 C_5HN	1+2	1	1	o
8	2,3,5,6-Cl_4 C_5HN	1+2	1	1 or A1	+
9	Cl_5C_5N	1+2	1	1	o
10	NaOH	SM	–	2	o
11	2-OH-6-Cl C_5H_3N	4+10	2	2	o
12	2-OH-C_5H_4N	3+10	2	2	o
13	2-OH-3,6-Cl_2 C_5H_2N	5+10	2	2	o
14	2-OH-5,6-Cl_2 C_5H_2N	5+10	2	2	o
15	2-OH-4,6-Cl_2 C_5H_2N	6+10	2	2	o
16	2-OH-3,4,6-Cl_3 C_5HN	7+10	2	2	o
17	2-OH-4,5,6-Cl_3 C_5HN	7+10	2	2	o
18	2-OH-3,5,6-Cl_3 C_5HN	2+11 8+10 35	3 2 21	3 A2 St	++
19	2-OH-3,4,5,6-Cl_4 C_5N	9+10	2	2	o
20		18+10	4	A2	+
21	C_2H_5OH	SM	–	4	o
22	P_2S_5	SM	–	4	o
23	$(C_2H_5O)_2P(S)SH$	21+22	5	4	o
24	$(C_2H_5O)_2P(S)SC_2H_5$	21+23 or 22+25	6 or 9	4	+
25	$(C_2H_5O)_3PS$	21+23 or 21+24	7 or 8	4	+
26	$(C_2H_5O)_2P(S)OH$	23 or 35	10 or 21	4 or St	+
27	$(C_2H_5O)_2P(O)SH$	26	11	4	+
28	$(C_2H_5O)_2P(S)S P(S)(OC_2H_5)_2$	23	12	4	o

TABLE B-14. (Continued)

Number	Contaminant(a)	Precursor(b)	Reaction Number	Step(c)	Likelihood of Being Present in Final Product(d)
29	$(C_2H_5O)_2P(S)SSP(S)(OC_2H_5)_2$	23	13 or 18	4	o
30	$(C_2H_5O)_2(C_2H_5S)PO$	25	14	4	o
31	$(C_2H_5O)_2PSH$	23	15	4	o
32	$(C_2H_5O)_3P$	31+21	16	4	o
33	$(C_2H_5O)_2(C_2H_5S)P$	31+21	17	4	+
34	$(C_2H_5O)_2P(S)Cl$	23+2	19	5	o
35	$(C_2H_5O)_2P(S)O$ 	18+34	20	6	**Chlorpyrifos**
36	$(C_2H_5O)_2P(S)O$ 	14+34	20	6	++
37	$(C_2H_5O)_2P(S)O$ 	13+34	20	6	++
38	$(C_2H_5O)_2P(S)O$ 	16+34	20	6	++
39	$(C_2H_5O)_2P(S)O$ 	17+34	20	6	++
40	$(C_2H_5O)_2P(S)O$ 	19+34	20	6	++
41	$(C_2H_5O)(C_2H_5S)P(O)O$ 	35	14	6 or St	++
42	$(C_2H_5O)_2P(O)S$ 	35	14	6 or St	+
43	$HCON(CH_3)_2$	Solv	–	6	u
44	$(C_2H_5O)_2P(O)O$ 	35	22	St	+
45	$C_2H_5OP(S)(OH)_2$	35	21	St	+
46	$2,5\text{-}(OH)_2\text{-}3,6\text{-}Cl_2C_5HN$	18	23	St	o
47	$2,3,5,6\text{-}(OH)_4C_5HN$	18	23	St	o
48		47	24	St	o

(a) Precursor refers to the contaminant numbered in this table. SM = starting material. Solv = solvent.

(b) See Table B-13.

(c) The step (Figures B-7 and B-8) during shich the contaminant might be formed. St indicates possible formation during storage.

(d) o = likely to be present at levels below 1 ppm
+ = likely to be present at levels above 1 ppm but below 100 ppm
++ = likely to be present at levels above 100 ppm.

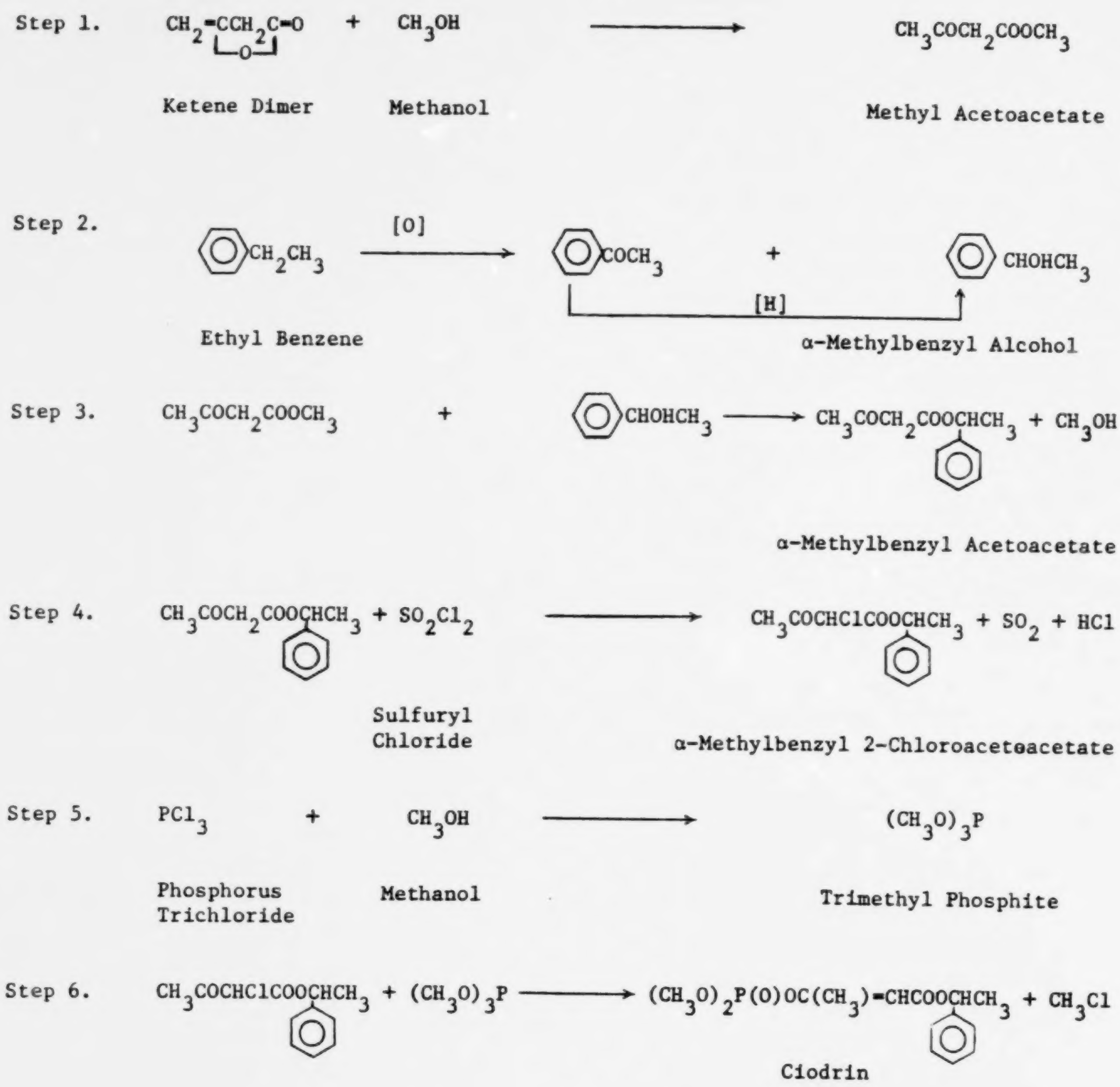

Step 1.
CH2=CCH2C=O
O
+
CH3OH
CH3COCH2COOCH3
Ketene Dimer
Methanol
Methyl Acetoacetate
Step 2.
CH2CH3
[O]
COCH3
+
CHOHCH3
[H]
Ethyl Benzene
α-Methylbenzyl Alcohol
Step 3.
CH3COCH2COOCH3
+
CHOHCH3
CH3COCH2COOCHCH3 + CH3OH
α-Methylbenzyl Acetoacetate
Step 4.
CH3COCH2COOCHCH3 + SO2Cl2
CH3COCHClCOOCHCH3 + SO2 + HCl
Sulfuryl Chloride
α-Methylbenzyl 2-Chloroacetoacetate
Step 5.
PCl3
+
CH3OH
(CH3O)3P
Phosphorus Trichloride
Methanol
Trimethyl Phosphite
Step 6.
CH3COCHClCOOCHCH3 + (CH3O)3P
(CH3O)2P(O)OC(CH3)=CHCOOCHCH3 + CH3Cl
Ciodrin

FIGURE B-9. SYNTHESIS OF CIODRIN

TABLE B-15. REACTIONS OCCURRING DURING PRODUCTION AND STORAGE OF CIODRIN

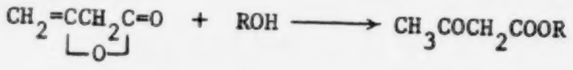
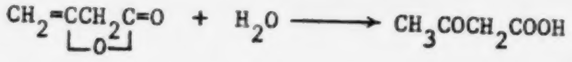
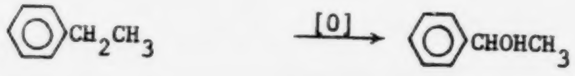
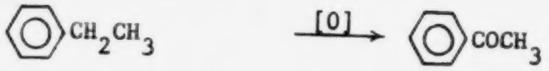
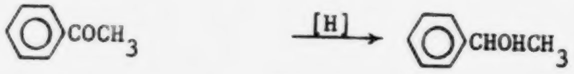
Reaction Number	Generalized Reaction
1.	 $CH_2{=}CCH_2C{=}O + ROH \longrightarrow CH_3COCH_2COOR$
2.	 $CH_2{=}CCH_2C{=}O + H_2O \longrightarrow CH_3COCH_2COOH$
3.	$CH_3COCH_2COOH \longrightarrow CH_3COCH_3 + CO_2$
4.	
5.	
6.	
7.	$RCOOR' + H_2O \rightleftharpoons RCOOH + ROH$
8.	$RCOOR' + R''OH \rightleftharpoons RCOOR'' + R'OH$
9.	$RCH_2COR' + SO_2Cl_2 \longrightarrow RCHClCOR' + SO_2 + HCl$
10.	$RCHClCOR + SO_2Cl_2 \longrightarrow RCCl_2COR'' + SO_2 + HCl$
11.	$ROH + PCl_3 \longrightarrow ROPCl_2 + HCl$
12.	$ROPCl_2 + ROH \longrightarrow (RO)_2PCl + HCl$
13.	$(RO)_2PCl + ROH \longrightarrow (RO)_3P + HCl$
14.	$(RO)_2P + HCl \longrightarrow ROP(OH)_2 + RCl$
15.	$(RO)_2POH + HCl \longrightarrow ROP(OH)_2 + RCl$

TABLE B-15. (Continued)

Reaction Number	Generalized Reaction
16.	$(RO)_3P \xrightarrow{[O]} (RO)_3PO$
17.	$(RO)_3P + R'CH_2COCHClR' \longrightarrow (RO)_2P(O)OC)R'CH_2)=CHR' + RCl$
18.	$(RO)_3PO + H_2O \longrightarrow (RO)_2P(O)OH + ROH$
19.	$(RO)_2P(O)OH + H_2O \longrightarrow ROP(O)(OH)_2 + ROH$
20.	$(RO)_2P(O)OC(R'CH_2)=CHR'' \xrightarrow{\Delta} (RO)_2P(O)OH + [R'CH=C=CHR'']_x$

TABLE B-16. POSSIBLE CONTAMINANTS IN CIODRIN

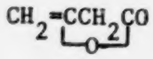
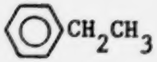
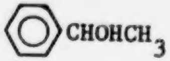
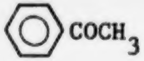
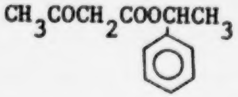
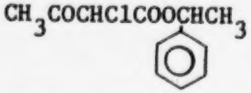
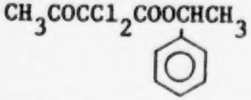
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	 $CH_2{=}CCH_2CO$	SM	–	1	0
2	CH_3OH	SM	–	1	0
3	$CH_3COCH_2COOCH_3$	1 + 2	1	1	0
4	CH_3COCH_2COOH	1	2	1	0
5	CH_3COCH_3	4 9	3 7 + 3	1 4	0 0
6		SM	–	2	0
7		6 8 9 26	4 6 7 7	2 2 4 St	++
8		6	5	2	
9		3 + 7	8	3	++
10	SO_2Cl_2	SM	–	4	0
11		9	9	4	++
12		11	10	4	+

TABLE B-16. (Continued)

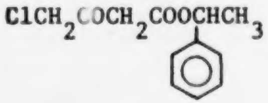
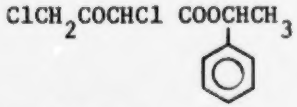
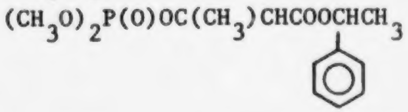
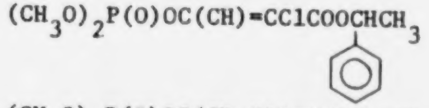
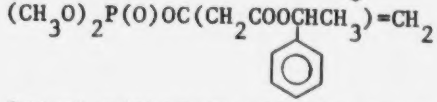
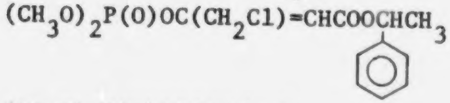
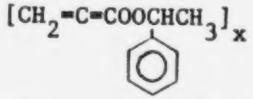
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
13	 $ClCH_2COCH_2COOCHCH_3$	9	9	4	+
14	 $ClCH_2COCHCl\ COOCHCH_3$	11	9	4	0
15	CH_3COCH_2Cl	5 13 31	9 7 18	4 6 St	+
16	$ClCH_2COCH_2Cl$	15	9	4	0
17	PCl_3	SM	-	5	0
18	$(CH_3O)PCl_2$	2 + 17	11	5	0
19	$(CH_3O)_2PCl$	18 + 2	12	5	0
20	$(CH_3O)_3P$	19 + 2	13	5	+
21	$(CH_3O)_2P(O)H$	20	14	5	+
22	$CH_3OP(OH)_2$	21	15	5	+
23	$P(OH)_3$	22	15	5	0
24	CH_3Cl	20 + 11	17	6	0
25	$(CH_3O)_3PO$	20	16	5	+
26	 $(CH_3O)_2P(O)OC(CH_3)CHCOOCHCH_3$	20 + 11	17	6	Ciodrin

TABLE B-16. (Continued)

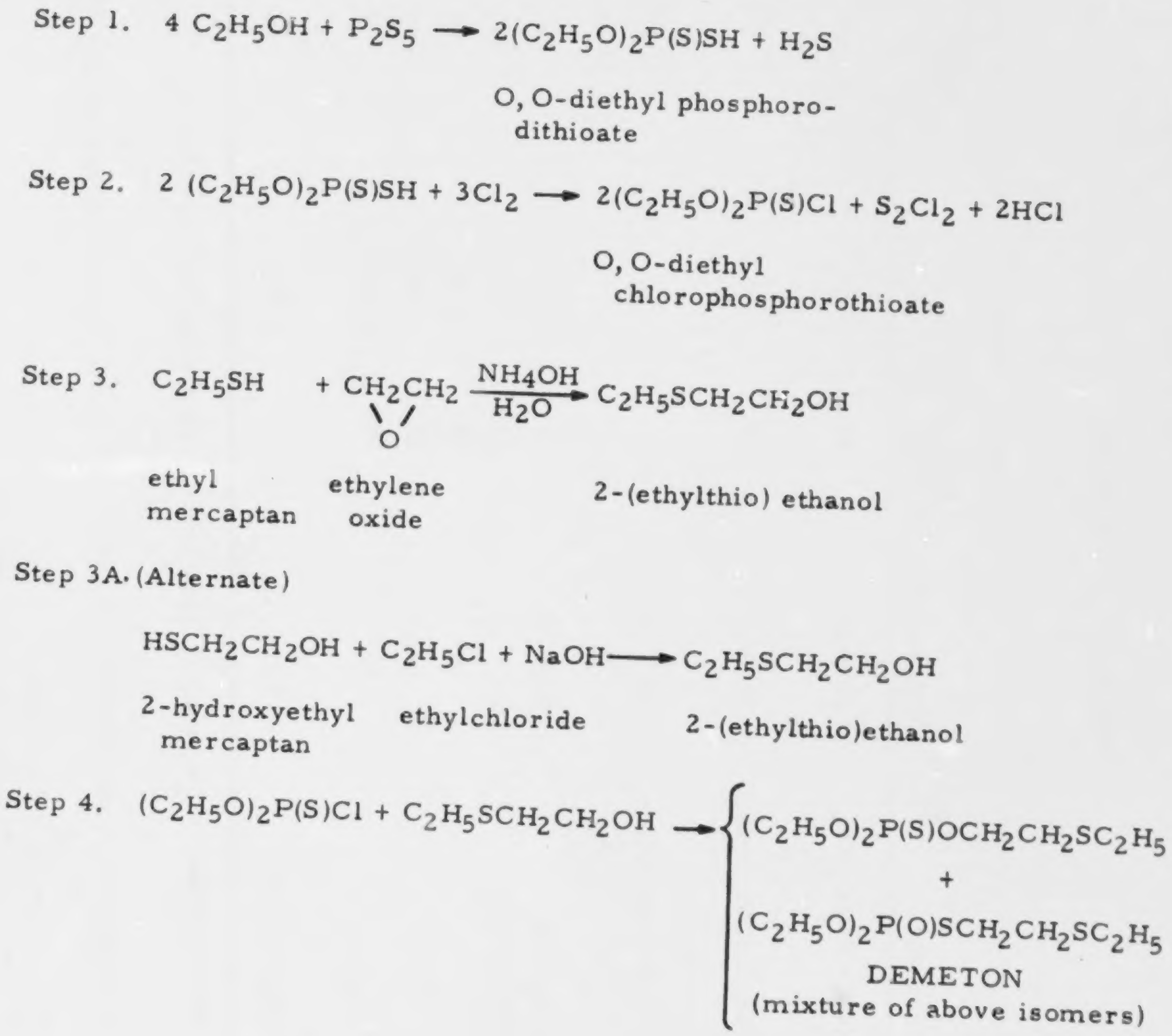
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
27	 $(CH_3O)_2P(O)OC(CH)=CClCOOCHCH_3$	20 + 12	17	6	+
28	 $(CH_3O)_2P(O)OC(CH_2COOCHCH_3)=CH_2$	20 + 13	17	6	+
29	 $(CH_3O)_2P(O)OC(CH_2Cl)=CHCOOCHCH_3$	20 + 14	17	6	0
30	$(CH_3O)_2P(O)OC(CH_3)=CH_2$	20 + 15	17	6	+
31	$(CH_3O)_2P(O)OC(CH_2Cl)=CH_2$	20 + 16	17	6	0
32	$(CH_3O)_2P(O)OH$	26	18	St	++
33	$CH_3OP(O)(OH)_2$	32	18	St	++
34	 $[CH_2=C=COOCHCH_3]_x$	26	20	6 + St	++
35	$(CH_3O)_2P(O)OC(CH_3)=CHCOOH$	26	7	6 + St	++

[a] Precursor refers to the contaminant numbered in this table. SM = starting material.

[b] See Table B-15.

[c] The step (Figure B-9) during which the contaminant might be formed. St indicates possible formation during storage.

[d] 0 = likely present only at levels below 1 ppm.
+ = likely present at levels above 1 ppm but below 100 ppm.
++= likely present at levels above 100 ppm.



Step 1. $4\ C_2H_5OH + P_2S_5 \rightarrow 2(C_2H_5O)_2P(S)SH + H_2S$

O, O-diethyl phosphorodithioate

Step 2. $2\ (C_2H_5O)_2P(S)SH + 3Cl_2 \rightarrow 2(C_2H_5O)_2P(S)Cl + S_2Cl_2 + 2HCl$

O, O-diethyl chlorophosphorothioate

Step 3. $C_2H_5SH + CH_2CH_2(O) \xrightarrow[H_2O]{NH_4OH} C_2H_5SCH_2CH_2OH$

ethyl mercaptan; ethylene oxide; 2-(ethylthio) ethanol

Step 3A. (Alternate)

$HSCH_2CH_2OH + C_2H_5Cl + NaOH \rightarrow C_2H_5SCH_2CH_2OH$

2-hydroxyethyl mercaptan; ethylchloride; 2-(ethylthio)ethanol

Step 4. $(C_2H_5O)_2P(S)Cl + C_2H_5SCH_2CH_2OH \rightarrow \left\{ \begin{array}{c} (C_2H_5O)_2P(S)OCH_2CH_2SC_2H_5 \\ + \\ (C_2H_5O)_2P(O)SCH_2CH_2SC_2H_5 \end{array} \right.$

DEMETON
(mixture of above isomers)

FIGURE B-10. SYNTHESIS OF DEMETON

TABLE B-17. REACTIONS OCCURRING DURING PRODUCTION OF DEMETON

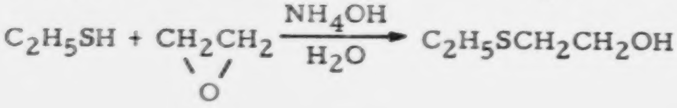
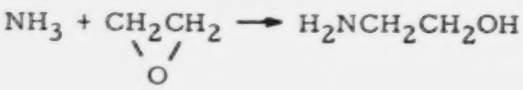
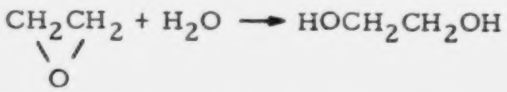
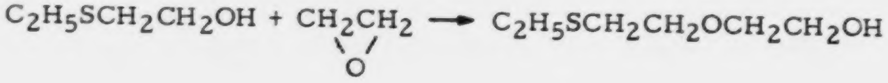
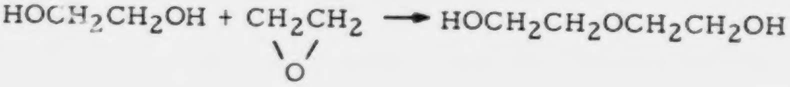
Number	Generalized Reaction[a]
1	$4C_2H_5OH + P_2S_5 \rightarrow 2(C_2H_5O)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightarrow (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)SH + H_2O \rightarrow (RY)_2P(Y)OH + H_2S$
6	$(RY)_2P(S)OH \rightleftarrows (RY)_2P(O)SH$
7	$2(RY)_2P(Y)YH \rightarrow (RY)_2P(Y)YP(Y)(YR)_2 + H_2Y$
8	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(YR)_2$
9	$2(RY)_2P(Y)SH + Cl_2 \rightarrow (RY)_2P(Y)SSP(Y)(YR)_2 + 2HCl$
10	$(RY)_2P(S)OR' \rightleftarrows (RY)_2P(O)SR'$
11	$(RY)_2P(S)SH \rightarrow (RY)_2PSH + S$
12	$(RY)_2PSH + R'OH \rightarrow (RY)_2POR' + H_2S$
13	$(RY)_2PSH + R'OH \rightarrow (RY)_2PSR' + H_2O$
14	$2(RY)_2P(S)SH + 3Cl_2 \rightarrow 2(RY)_2P(S)Cl + S_2Cl_2 + HCl$
15	$(RY)_2P(S)OH + (RY)_2P(S)Cl \rightarrow (RY)_2P(S)OP(S)(YR)_2 + HCl$
16	$(RY)_2P(O)SH + (RY)_2P(S)Cl \rightarrow (RY)_2P(O)SP(S)(YR)_2 + HCl$
17	 $C_2H_5SH + \underset{\setminus\;O\;/}{CH_2CH_2} \xrightarrow[H_2O]{NH_4OH} C_2H_5SCH_2CH_2OH$
18	 $NH_3 + \underset{\setminus\;O\;/}{CH_2CH_2} \rightarrow H_2NCH_2CH_2OH$
19	 $\underset{\setminus\;O\;/}{CH_2CH_2} + H_2O \rightarrow HOCH_2CH_2OH$

TABLE B-17. (Continued)

Number	Generalized Reaction(a)
20	 $C_2H_5SCH_2CH_2OH + \underset{O}{CH_2CH_2} \rightarrow C_2H_5SCH_2CH_2OCH_2CH_2OH$
21	 $HOCH_2CH_2OH + \underset{O}{CH_2CH_2} \rightarrow HOCH_2CH_2OCH_2CH_2OH$
22	$2RSH \xrightarrow{[O]} RSSR$
23	$(C_2H_5O)_2P(S)Cl + R''YH \rightarrow (C_2H_5O)_2P(S)OR''$
24	$(C_2H_5O)_2P(S)Cl + H_2NCH_2CH_2OH \rightarrow (C_2H_5O)_2P(S)NHCH_2CH_2OH$
25	$(C_2H_5O)_2P(Y)YCH_2CH_2SC_2H_5 \xrightarrow{[O]} (C_2H_5O)_2P(Y)YCH_2CH_2SOC_2H_5$
26	$(C_2H_5O)_2P(Y)YCH_2CH_2SOC_2H_5 \xrightarrow{[O]} (C_2H_5O)_2P(Y)YCH_2CH_2SO_2C_2H_5$
27	$(C_2H_5O)_2P(S)OCH_2CH_2SC_2H_5 \xrightarrow{[O]} (C_2H_5O)_2P(O)OCH_2CH_2SC_2H_5$
28	$(C_2H_5O)_2P(S)OCH_2CH_2SC_2H_5 + H_2O \rightarrow (C_2H_5O)_2P(S)OH$ $+ C_2H_5OP(S)(OH)_2$ $+ C_2H_5SCH_2CH_2OH$ $+ C_2H_5OH$
29	$(C_2H_5O)_2P(O)SCH_2CH_2SC_2H_5 + H_2O \rightarrow (C_2H_5O)_2P(O)SH$ $+ (C_2H_5O)_2P(O)OH$ $+ C_2H_5SCH_2CH_2OH$ $+ C_2H_5SCH_2CH_2SH$

(a) R and R' = alkyl or substituted alkyl
R'' = $C_2H_5SCH_2CH_2-$, $C_2H_5SCH_2CH_2OCH_2CH_2-$,
$HOCH_2CH_2-$ or $HOCH_2CH_2OCH_2CH_2-$
Y = oxygen or sulfur.

TABLE B-18. POSSIBLE CONTAMINANTS IN DEMETON

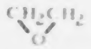
Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
1	C_2H_5OH	SM	--	1	0
2	P_2S_5	SM	--	1	0
3	$(C_2H_5O)_2P(S)SH$	1 + 2	1	1	0
4	$(C_2H_5O)_2P(S)SC_2H_5$	1 + 3	2	1	+
5	$(C_2H_5O)_3PS$	1 + 3 1 + 4	3 4	1	+
6	$(C_2H_5O)_2P(S)OH$	3 27	5 28	1 St	+
7	$(C_2H_5O)_2P(O)SH$	6 28	6 29	1 St	+
8	$(C_2H_5O)_2P(S)SP(S)(OC_2H_5)_2$	3	7	1	0
9	$(C_2H_5O)_2P(S)SSP(S)(OC_2H_5)_2$	3 3	8 9	1 2	+
10	$(C_2H_5O)_2(C_2H_5S)PO$	5	10	1	0
11	$(C_2H_5O)_2PSH$	3	11	1	0
12	$(C_2H_5O)_3P$	1 + 11	12	1	0
13	$(C_2H_5O)_2(C_2H_5S)P$	1 + 11	13	1	+
14	Cl_2	SM	--	2	0
15	$(C_2H_5O)_2P(S)Cl$	3 + 14	14	2	+
16	$(C_2H_5O)_2P(S)OP(S)(OC_2H_5)_2$	6 + 15	15	2	++
17	$(C_2H_5O)_2P(O)SP(S)(OC_2H_5)_2$	7 + 15	16	2	++
18	C_2H_5SH	SM	--	3	0
19		SM	--	3	0
20	$C_2H_5SCH_2CH_2OH$	18 + 19 27	17 28	3 St	++
21	NH_3	Cat.	--	3	0
22	$H_2NCH_2CH_2OH$	19 + 21	18	3	0
23	$HOCH_2CH_2OH$	19	19	3	0
24	$C_2H_5SCH_2CH_2OCH_2CH_2OH$	19 + 20	20	3	+
25	$HOCH_2CH_2OCH_2CH_2OH$	19 + 23	21	3	0
26	$C_2H_5SSC_2H_5$	18	22	3	0

TABLE B-18. (Continued)

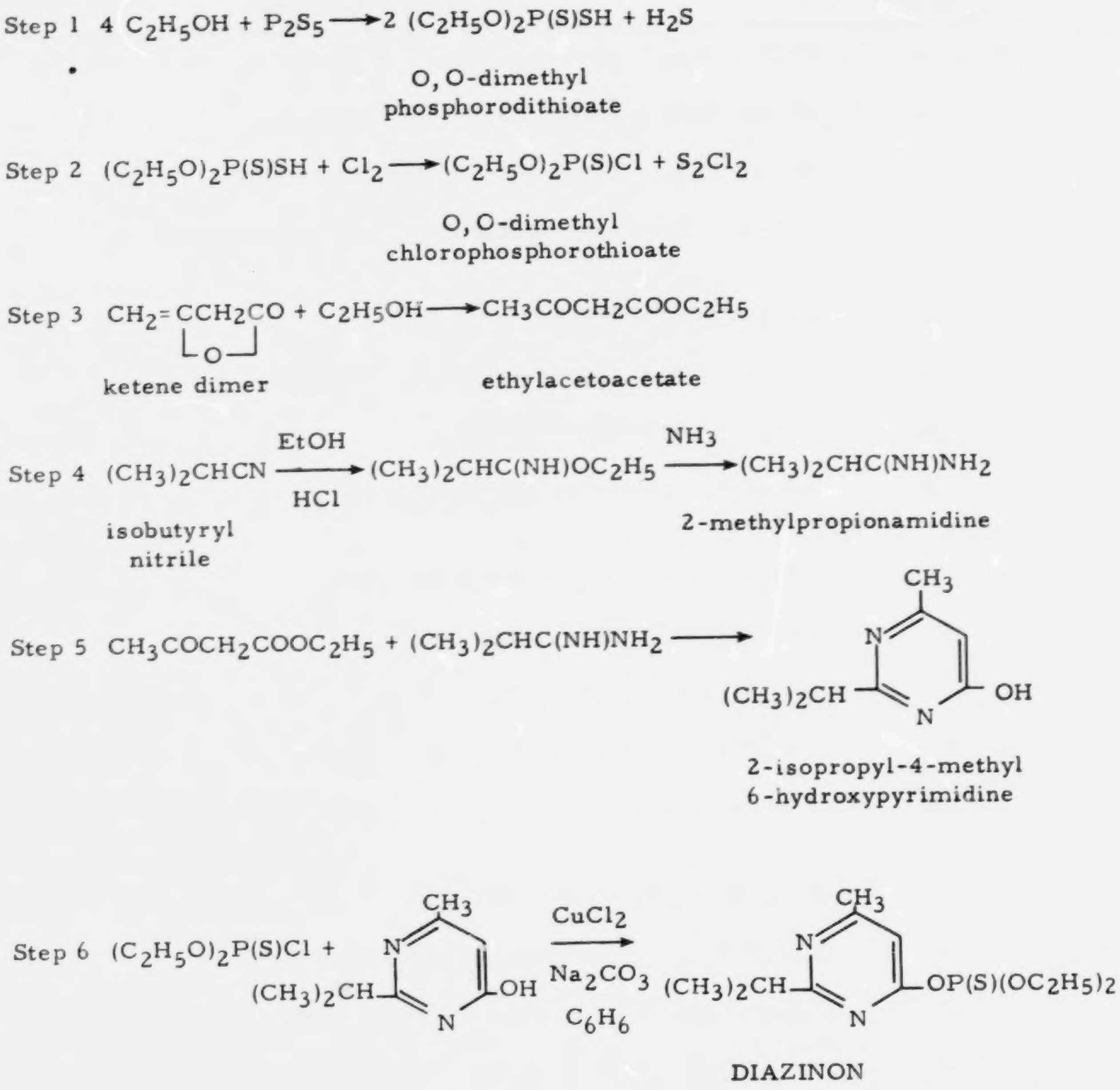
Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
27	$(C_2H_5O)_2P(S)OCH_2CH_2SC_2H_5$	15 + 20	23	4	DEMETON (mixture of 27 and 28)
28	$(C_2H_5O)_2P(O)SCH_2CH_2SC_2H_5$	27	10	4 or St	
29	$(C_2H_5O)(C_2H_5S)P(O)OCH_2CH_2SC_2H_5$	27	10	4	+
30	$(C_2H_5O)_2P(S)OCH_2CH_2OCH_2CH_2SC_2H_5$	15 + 24	23	4	+
31	$(C_2H_5O)_2P(O)SCH_2CH_2OCH_2CH_2SC_2H_5$	30	10	4	+
32	$(C_2H_5O)_2P(S)OCH_2CH_2OH$	15 + 23	23	4	0
33	$(C_2H_5O)_2P(S)OCH_2CH_2OCH_2CH_2OH$	15 + 25	23	4	0
34	$(C_2H_5O)_2P(O)SCH_2CH_2OH$	32	10	4	0
35	$(C_2H_5O)_2P(S)NHCH_2CH_2OH$	15 + 22	24	4	0
36	$HSCH_2CH_2OH$	SM	--	3A	+
37	C_2H_5Cl	SM	--	3A	0
38	$(C_2H_5O)_2P(S)SCH_2CH_2OH$	15 + 36	23	4	+
39	$(C_2H_5O)_2P(S)OCH_2CH_2SOC_2H_5$	27	25	St	+
40	$(C_2H_5O)_2P(S)OCH_2CH_2SO_2C_2H_5$	39	26	St	+
41	$(C_2H_5O)_2P(O)SCH_2CH_2SOC_2H_5$	28	25	St	+
42	$(C_2H_5O)_2P(O)SCH_2CH_2SO_2C_2H_5$	41	26	St	+
43	$(C_2H_5O)_2P(O)OCH_2CH_2SC_2H_5$	27	27	St	+
44	$C_2H_5OP(S)(OH)_2$	27	28	St	+
45	$(C_2H_5O)_2P(O)OH$	28	29	St	+
46	$C_2H_5SCH_2CH_2SH$	28	29	St	+
47	$C_2H_5SCH_2CH_2SSCH_2CH_2SC_2H_5$	46	22	St	+

[a] Precursor refers to the contaminant numbered in this table.
SM starting material
Cat. = catalyst

[b] See Table B-17.

[c] The step (Figure B-10) during which the contaminant might be formed; St indicates possible formation during storage.

[d] 0 likely to be present at levels below 1 ppm
+ = likely to be present at levels above 1 ppm but below 100 ppm
++ = likely to be present at levels above 100 ppm.



Step 1 $4\ C_2H_5OH + P_2S_5 \longrightarrow 2\ (C_2H_5O)_2P(S)SH + H_2S$

O, O-dimethyl phosphorodithioate

Step 2 $(C_2H_5O)_2P(S)SH + Cl_2 \longrightarrow (C_2H_5O)_2P(S)Cl + S_2Cl_2$

O, O-dimethyl chlorophosphorothioate

Step 3 $CH_2{=}CCH_2CO + C_2H_5OH \longrightarrow CH_3COCH_2COOC_2H_5$

ketene dimer

ethylacetoacetate

Step 4 $(CH_3)_2CHCN \xrightarrow[HCl]{EtOH} (CH_3)_2CHC(NH)OC_2H_5 \xrightarrow{NH_3} (CH_3)_2CHC(NH)NH_2$

isobutyryl nitrile

2-methylpropionamidine

Step 5 $CH_3COCH_2COOC_2H_5 + (CH_3)_2CHC(NH)NH_2 \longrightarrow$

CH3
N
(CH3)2CH
N
OH

2-isopropyl-4-methyl 6-hydroxypyrimidine

Step 6 $(C_2H_5O)_2P(S)Cl +$

CH3
N
(CH3)2CH
N
OH
CuCl2
Na2CO3
C6H6
CH3
N
(CH3)2CH
N
OP(S)(OC2H5)2

DIAZINON

FIGURE B-11. SYNTHESIS OF DIAZINON

TABLE B-19. REACTIONS OCCURRING DURING PRODUCTION AND STORAGE OF DIAZINON

Reaction Number	Generalized Reaction
1.	$4ROH + P_2S_5 \rightarrow 2(RO)_2P(S)SH + H_2S$
2.	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)SR' + H_2O$
3.	$(RY)_2P(Y)SH + R'OH \rightarrow (RY)_2P(Y)OR' + H_2S$
4.	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5.	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6.	$(RY)_2P(Y)SH + H_2O \rightarrow (RY)_2P(Y)OH + H_2S$
7.	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
8.	$(RY)_2P(Y)YH \rightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
9.	$(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
10.	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
11.	$(RY)_2P(S)SH \rightarrow (RY)_2PSH + S$
12.	$(RY)_2PSH + R'OH \rightarrow (RY)_2POR' + H_2S$
13.	$(RY)_2PSH + R'OH \rightarrow (RY)_2PSR' + H_2O$
14.	$(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(YR)_2$
15.	$(RY)_2PSH + Cl_2 \rightarrow (RY)_2P(S)SP(YR)_2 + 2HCl$
16.	$(RY)_2P(S)SH + Cl_2 \rightarrow (RY)_2P(S)SSP(S)(YR)_2 + 2HCl$
17.	$(RY)_2P(S)SSP(S)(YR)_2 + 2Cl_2 \rightarrow 2(RY)_2P(S)Cl + S_2Cl_2$
18.	$2S_2Cl_2 + 2H_2O \rightarrow SO_2 + 4HCl + S$
19.	$RSH \xrightarrow{[O]} RSSR$

TABLE B-19. (Continued)

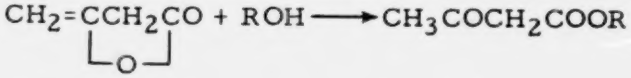
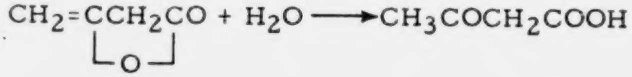
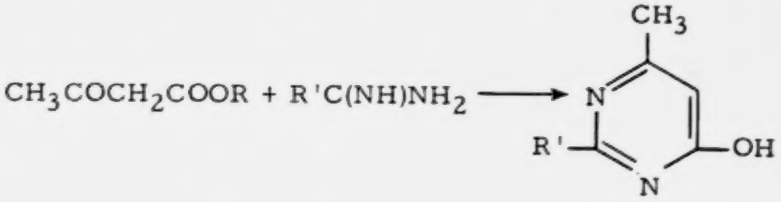
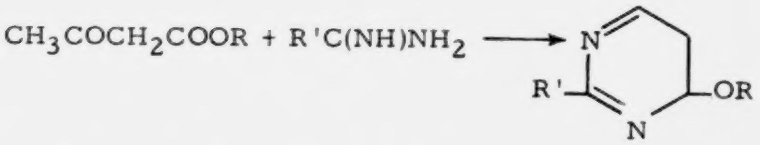
Reaction Number	Generalized Reaction
20.	 $CH_2{=}\underbrace{CCH_2CO}_{O} + ROH \longrightarrow CH_3COCH_2COOR$
21.	 $CH_2{=}\underbrace{CCH_2CO}_{O} + H_2O \longrightarrow CH_3COCH_2COOH$
22.	$CH_3COCH_2COOH \longrightarrow CH_3COCH_3 + CO_2$
23.	$RCN + R'OH \xrightarrow{HCl} RC(NH)OR' + HCl$
24.	$RC(NH)OR' + NH_3 \longrightarrow RC(NH)NH_2 + R'OH$
25.	$RC(NH)OR' + H_2O \longrightarrow RCONH_2 + R'OH$
26.	
27.	
28.	$(RY)_2P(S)Cl + R'OH \longrightarrow (RY)_2P(S)OR'$
29.	$(RY)_2P(S)Cl + NH_3 \longrightarrow (RY)_2P(S)NH_2$
30.	$(RY)_2P(S)Cl + R'C(NH)NH_2 \longrightarrow (RY)_2P(S)NHC(NH)R'$
31.	$(RY)_3P(Y) + R'OH \longrightarrow (RY)_2P(Y)OR'\ RYH$
32.	$(RY)_2P(Y)Cl + Na_2CO_3 \longrightarrow (RY)_2P(Y)YNa$
33.	$(RY)_2P(Y)Cl + (RY)_2P(Y)YNa \longrightarrow (RY)_2P(Y)YP(Y)(YR)_2$
34.	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
35.	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$

TABLE B-20. POSSIBLE CONTAMINANTS IN DIAZINON

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product[(d)]
1	P_2S_5	SM	–	1	0
2	$(C_2H_5O)_2P(S)SH$	1	1	1	+
3	$(C_2H_5O)_2(C_2H_5S)PS$	2	2	1	++
4	$(C_2H_5O)_3PS$	2 3	3 4	1 1	+
5	$(C_2H_5O)(C_2H_5S)_2PS$	3	5	1	++
6	$(C_2H_5O)(C_2H_5S)P(S)SH$	2	5	1	0
7	$(C_2H_5O)_2P(S)OH$	2 35	6 35	1 St	+
8	$(C_2H_5O)_2P(O)OH$	7			+
9	$(C_2H_5O)(C_2H_5S)P(O)SH$	6	6+7	1	0
10	$(C_2H_5O)_2P(S)SP(S)(OC_2H_5)_2$	2	8	1	++
11	$(C_2H_5O)_2P(S)SSP(S)(OC_2H_5)_2$	2 2	9 16	1 2	+
12	$(C_2H_5O)_2PSH$	2	11	1	0

TABLE B-20. (Continued)

No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
13	$(C_2H_5O)_3P$	12	12	1	0
14	$(C_2H_5O)_2(C_2H_5S)P$	12	13	1	0
15	$(C_2H_5O)_2P(S)SP(OC_2H_5)_2$	12 12	14 15	1 2	0
16	$(C_2H_5O)_2(C_2H_5S)PO$	4 9	10 3	1 1	++
17	$(C_2H_5O)_3PO$	16	4	1	0
18	$(C_2H_5S)_3PS$	5	5	1	+
19	$(C_2H_5O)(C_2H_5S)_2PO$	3	10	1	++
20	$(C_2H_5S)_3PO$	5	10	1	+
21	$(C_2H_5O)_2P(S)Cl$	11	17	2	+
22	$(C_2H_5O)(C_2H_5S)P(S)Cl$	6	16+17	2	0
23	S_2Cl_2	11	17	2	0
24	S	23	18	2	+

TABLE B-20. (Continued)

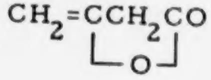
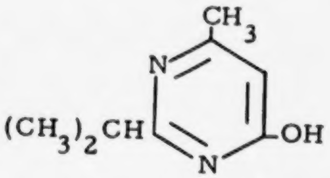
No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product[(d)]
25	 $CH_2=CCH_2CO$, O	SM	—	3	0
26	$CH_3COCH_2COOC_2H_5$	25	20	3	0
27	CH_3COCH_2COOH	25	21	3	+
28	CH_3COCH_3	27	22	3	0
29	$(CH_3)_2CHCN$	SM	–	4	+
30	$(CH_3)_2CHC(NH)OC_2H_5$	29	23	4	+
31	$(CH_3)_2CHC(NH)NH_2$	30	24	4	+
32	$(CH_3)_2CHCONH_2$	30	25	4	+
33		31+26	26	5	++

TABLE B-20. (Continued)

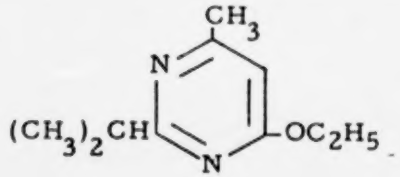
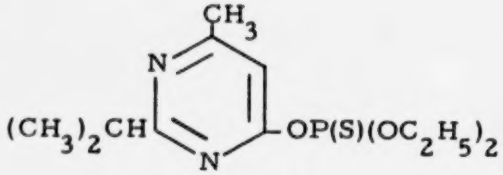
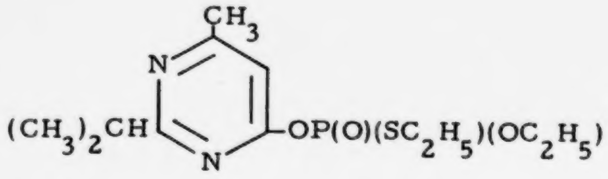
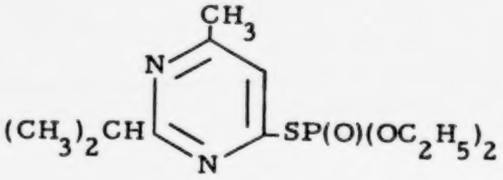
No.	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
34		31+26	27	5	++
35		33+21	28	6	Diazinon
36		35	10	6 or St	++
37		35	10	6 or St	+

TABLE B-20. (Continued)

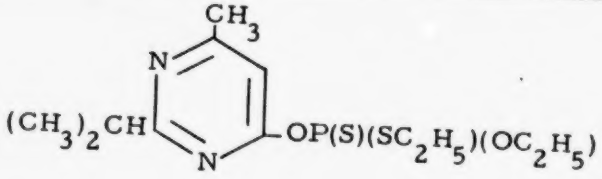
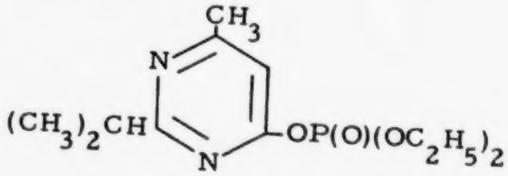
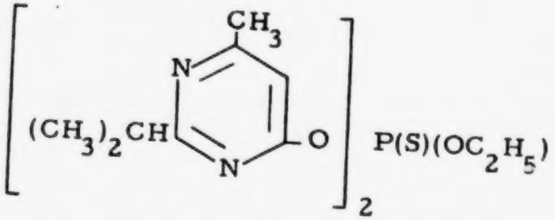
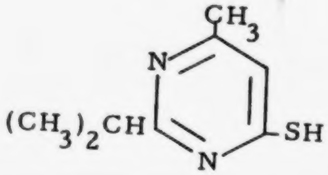
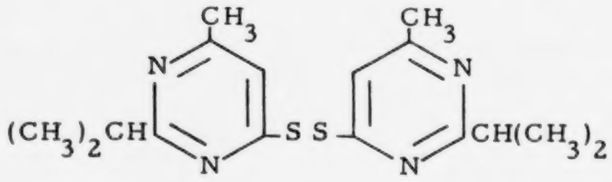
No.	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
38		33+22	28	6	++
39		35	34	6 or St	+
40		35+33	31	6	+
41		37	35 31	6 6	+

TABLE B-20. (Continued)

No.	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
42	$(C_2H_5O)_2P(S)ONa$	21	32	6	+
43	$(C_2H_5O)_2P(S)OP(S)(OC_2H_5)_2$	42+21	33	6	+
44		41	19	6	0
45	C_2H_5OH	35	35	6 or St	+
46	C_2H_5SH	36	35	6 or St	+
47	$C_2H_5SSC_2H_5$	46	19	6 or St	+
48	$(C_2H_5O)_2P(S)NH_2$	21	29	6	+
49	$(C_2H_5O)_2P(S)NHC(NH)CH(CH_3)_2$	21+31	30	6	++

(a) Precursor refers to the contaminant numbered in this table. SM = starting material.

(b) **See Table B-19**

(c) **The step (Figure B-11) during which the contaminant might be formed. St indicates possible formation during storage.**

(d) 0 = Likely to be present only at levels below 1 ppm
+ = Likely to be present at levels above 1 ppm but below 100 ppm.
++ = Likely to be present at levels above 100 ppm.

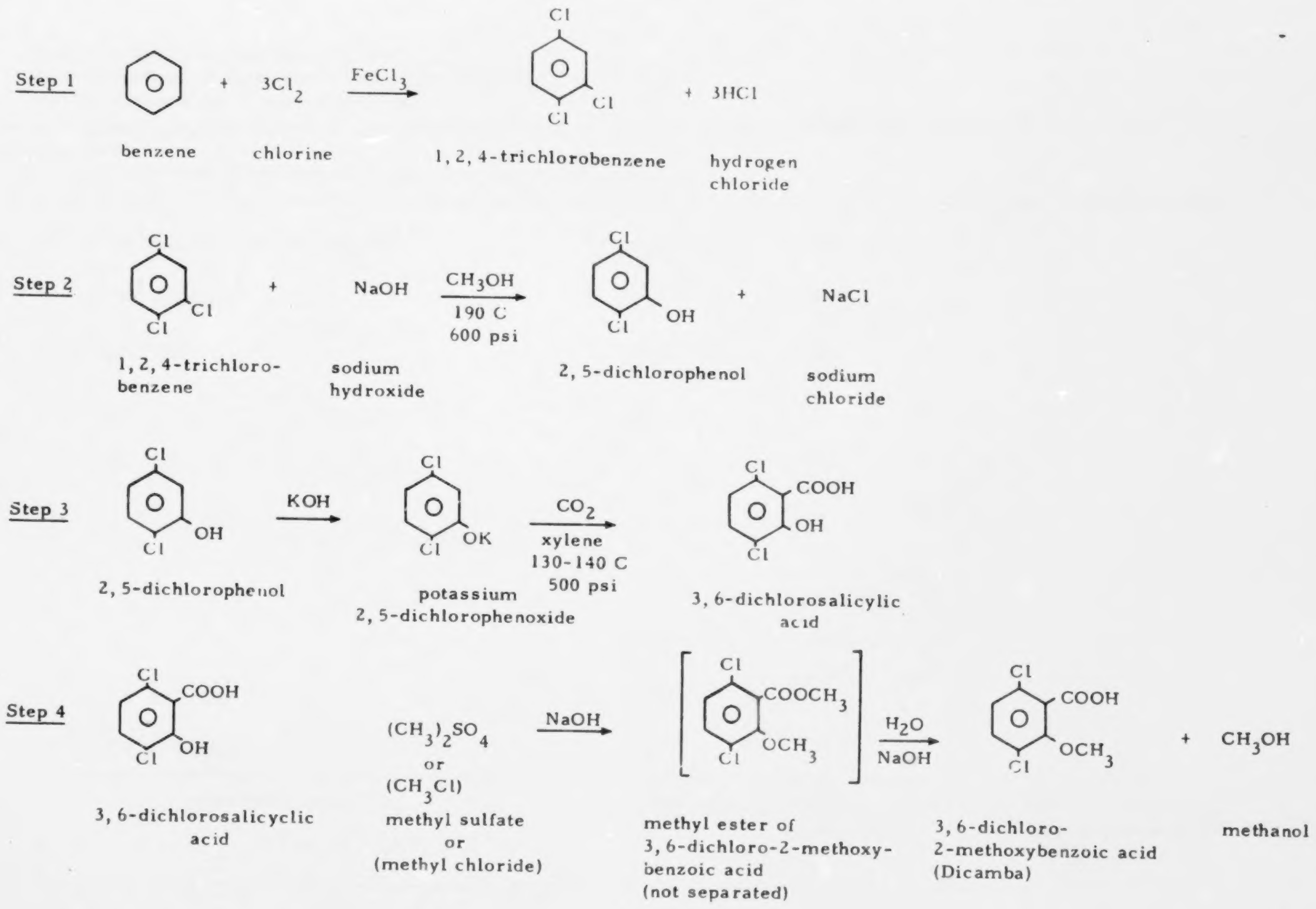

Step 1
+
$3Cl_2$
$FeCl_3$
Cl
Cl
Cl
+ 3HCl
benzene
chlorine
1, 2, 4-trichlorobenzene
hydrogen chloride
Step 2
Cl
Cl
Cl
+
NaOH
CH_3OH
190 C
600 psi
Cl
Cl
OH
+
NaCl
1, 2, 4-trichloro-benzene
sodium hydroxide
2, 5-dichlorophenol
sodium chloride
Step 3
Cl
Cl
OH
KOH
Cl
Cl
OK
CO_2
xylene
130-140 C
500 psi
Cl
Cl
COOH
OH
2, 5-dichlorophenol
potassium 2, 5-dichlorophenoxide
3, 6-dichlorosalicylic acid
Step 4
Cl
Cl
COOH
OH
$(CH_3)_2SO_4$
or
(CH_3Cl)
NaOH
Cl
Cl
$COOCH_3$
OCH_3
H_2O
NaOH
Cl
Cl
COOH
OCH_3
+
CH_3OH
3, 6-dichlorosalicyclic acid
methyl sulfate
or
(methyl chloride)
methyl ester of
3, 6-dichloro-2-methoxy-benzoic acid
(not separated)
3, 6-dichloro-2-methoxybenzoic acid
(Dicamba)
methanol

FIGURE B-12. SYNTHESIS OF DICAMBA

TABLE B-21. REACTIONS OCCURRING DURING PRODUCTION OF DICAMBA

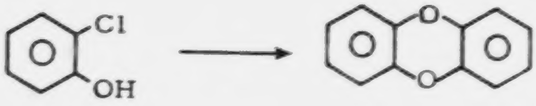
Reaction Number	Generalized Reaction
1	$ArH + Cl_2 \xrightarrow{FeCl_3} ArCl + HCl$
2	$ArCl + NaOH \longrightarrow ArOH + NaCl$
3	$ArCl + Ar'OH \xrightarrow{NaOH} ArOAr' + NaCl$
4	2 
5	$ArOH \xrightarrow{KOH} ArOK \xrightarrow{CO_2} Ar(OH)COOH$
6	$ArCOOH \xrightarrow[NaOH]{(CH_3)_2SO_4} ArCOOCH_3$
7	$ArOH \xrightarrow[NaOH]{(CH_3)_2SO_4} ArOCH_3$
8	$ArCOOCH_3 + H_2O \longrightarrow ArCOOH + CH_3OH$

TABLE B-22. POSSIBLE CONTAMINANTS IN DICAMBA

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	Benzene	SM	--	1	0
2	C_6H_5Cl	1	1	1	0
3	$C_6H_4Cl_2$	2	1	1	0
4	$C_6H_3Cl_3$	3	1	1	+
5	$C_6H_2Cl_4$	4	1	1	0
6	C_6HCl_5	5	1	1	0
7	C_6Cl_6	6	1	1	0
8	C_6H_5OH	2	2	2	0
9	C_6H_4ClOH	3	2	2	+
10	$C_6H_3Cl_2OH$	4	2	2	++
11	$C_6H_2Cl_3OH$	5	2	2	+
12	C_6HCl_4OH	6	2	2	+
13	C_6Cl_5OH	7	2	2	0
14	$C_6H_4(OH)_2$	9	2	2	0
15	$C_6H_3Cl(OH)_2$	10	2	2	0
16	$C_6H_2Cl_2(OH)_2$	11	2	2	0
17	$C_6HCl_3(OH)_2$	12	2	2	0
18	$C_6Cl_4(OH)_2$	13	2	2	0
19	$ClC_6H_4OC_6H_4Cl$	3+9	3	2	0
20	$Cl_2C_6H_3OC_6H_4Cl$	4+9 10+3	3	2	+
21	$Cl_2C_6H_3OC_6H_3Cl_2$	4+10	3	2	+

TABLE B-22. (Continued)

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
22	$Cl_3C_6H_2OC_6H_4Cl$	5+9 11+3	3	2	0
23	$Cl_3C_6H_2OC_6H_3Cl_2$	5+10 11+4	3	2	+
24	$Cl_3C_6H_2OC_6H_2Cl_3$	5+11	3	2	0
25	$Cl_4C_6HOC_6H_3Cl_2$	6+10 12+4	3	2	0
26	$Cl_5C_6OC_6H_3Cl_2$	7+10 13+4	3	2	0
27	Cl_2-DBD[e]	9+11 10	4	2	0[f]
28	Cl_3-DBD[e]	10+11	4	2	0[f]
29	Cl_4-DBD[e]	10+12 11	4	2	0[f]
30	$ClC_6H_3(OH)COOH$	9	5	3	+
31	$Cl_2C_6H_2(OH)COOH$	10	5	3	++
32	$Cl_3C_6H(OH)COOH$	11	5	3	+
33	$Cl_4C_6(OH)COOH$	12	5	3	0
34	$ClC_6H(OH)_2COOH$	15	5	3	0
35	$ClC_6H_3(OH)COOCH_3$	30	6	4	0
36	$Cl_2C_6H_2(OH)COOCH_3$	31	6	4	0
37	$Cl_3C_6H(OH)COOCH_3$	32	6	4	0
38	$Cl_4C_6(OH)COOCH_3$	33	6	4	0
39	$ClC_6H(OH)_2COOCH_3$	34	6	4	0
40	$ClC_6H_3(OCH_3)COOCH_3$	35	7	4	+

TABLE B-22. (Continued

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
41	$Cl_2C_6H_2(OCH_3)COOCH_3$	36	7	4	++
42	$Cl_3C_6H(OCH_3)COOCH_3$	37	7	4	+
43	$Cl_4C_6(OCH_3)COOCH_3$	38	7	4	0
44	$ClC_6H(OH)(OCH_3)COOCH_3$	39	7	4	0
45	$ClC_6H(OCH_3)_2COOCH_3$	44	7	4	0
46	$ClC_6H_3(OCH_3)COOH$	40	8	4	++
47	$Cl_2C_6H_2(OCH_3)COOH$	41	8	4	DICAMBA
48	$Cl_3C_6H(OCH_3)COOH$	42	8	4	++
49	$Cl_4C_6(OCH_3)COOH$	43	8	4	+
50	$ClC_6H(OH)(OCH_3)COOH$	44	8	4	0
51	$ClC_6H(OCH_3)_2COOH$	45	8	4	+

a. Precursor refers to the contaminant numbered in this table. SM = starting material. Solv. = solvent.

b. See Table B-21.

c. The step (see Figure B-12) during which the contaminant might be formed.

d. 0 = likely to be present at levels below 1 ppm.
+ = likely to be present at levels above 1 ppm but below 100 ppm.
++ = likely to be present at levels above 100 ppm.

e. DBD = Dibenzo-p-dioxin

f. Analysis of dicamba by E. A. Woolson, et. al. (J. Agr. and Food Chem. 20 (2), 351-354 (1972) showed <0.5 ppm of any one chlorodioxin in this pesticide.

Step 1. $4CH_3OH + P_2S_5 \longrightarrow 2(CH_3O)_2P(S)SH + H_2S$

0,0-dimethyl
phosphorodithioate

Step 2. $ClCH_2COOH + CH_3OH \xrightarrow{H^+} ClCH_2COOCH_3 + H_2O$

chloroacetic
acid

methyl
chloroacetate

Step 3. $ClCH_2COOCH_3 + CH_3NH_2 \longrightarrow ClCH_2CONHCH_3 + CH_3OH$

methylamine

N-methylchloroacetamide

Step 4. $(CH_3O)_2P(S)SH + ClCH_2CONHCH_3 \xrightarrow[\text{solvent*}]{\text{alkali or tertiary amine}} (CH_3O)_2P(S)SCH_2CONHCH_3$

O,O-dimethyl
S-(N-methylcarbamoylmethyl)
phosphorodithioate

(Dimethoate)

---

*Solvents include: chloroform, methylene chloride, toluene, benzene, carbon tetrachloride, and cyclohexanone.

FIGURE B-13. SYNTHESIS OF DIMETHOATE

Alternative A

Step A-1. Same as Step 1.

Step A-2. Same as Step 2.

Step A-3.

$$(CH_3O)_2P(S)SH + ClCH_2COOCH_3 \longrightarrow (CH_3O)_2P(S)SCH_2COOCH_3$$

O,O-dimethyl phosphorodithioate + methyl chloroacetate → O,O-dimethyl S-(carbomethoxymethyl) phosphorodithioate

Step A-4.

$$(CH_3O)_2P(S)SCH_2COOCH_3 + CH_3NH_2 \xrightarrow{CH_3OH} (CH_3O)_2P(S)SCH_2CONHCH_3$$

methylamine

methanol

Dimethoate

Alternative B

Step B-1. Same as Step 1.

Step B-2.

$$(CH_3O)_2P(S)SH + ClCH_2COOH \xrightarrow{\text{alkali}} (CH_3O)_2P(S)SCH_2COOH$$

O,O-dimethyl phosphorodithioate + chloroacetic acid → O,O-dimethyl S-(carboxymethyl) phosphorodithioate

Step B-3.

$$(CH_3O)_2P(S)SCH_2COOH + CH_3NCO \xrightarrow{C_6H_6} (CH_3O)_2P(S)SCH_2CONHCH_3 + CO_2$$

methyl isocyanate

Dimethoate

benzene

FIGURE B-14. ALTERNATIVE SYNTHESES OF DIMETHOATE

TABLE B-23. REACTIONS OCCURRING DURING PRODUCTION AND STORAGE OF DIMETHOATE

Reaction Number	Generalized Reaction(a)
1	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6	$(RO)_2P(Y)SH \xrightarrow{P_2S_5} (RO)(RS)P(Y)SH + (RS)_2P(Y)SH$
7	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
8	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$
9	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
10	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
11	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
12	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
13	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
14	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
15	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(RY)_2$
16	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2(R'S)PO$
17	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
18	$Cl_xCH_{(3-x)}COOH + CH_3OH \longrightarrow Cl_xCH_{(3-x)}COOCH_3 + H_2O$ $x = 1, 2,$ or 3
19	$ClCH_2COOCH_3 + R''R'''NH \longrightarrow ClCH_2CONR''R''' + CH_3OH$
20	$Cl_2CHCOOCH_3 + R''R'''NH \longrightarrow Cl_2CHCONR''R''' + CH_3OH$
21	$ClCH_2CONR''R''' + R''R'''NH \longrightarrow R''R'''NCH_2CONR''R'''$
22	$CH_3NHCH_2CONR''R''' + ClCH_2CONR''R''' \longrightarrow CH_3N(CH_2CONR''R''')_2$

TABLE B-23. (Continued)

Reaction Number	Generalized Reaction(a)
23	$(RY)_2P(S)SH + ClCH_2COZ \xrightarrow{\text{alkali}} (RY)_2P(S)SCH_2COZ$
24	$2(RY)_2P(S)SH + Cl_2CHCOZ \xrightarrow{\text{alkali}} [(RY)_2P(S)S]_2CHCOZ$
25	$CH_3OH + ClCH_2COZ \longrightarrow CH_3OCH_2COZ$
26	$(CH_3O)_2P(S)SCH_2CONHCH_3 + H_2O \xrightarrow{OH^-} (CH_3O)_2P(S)OH + HSCH_2CONHCH_3$
27	$(CH_3O)_2P(S)SCH_2CONHCH_3 + H_2O \xrightarrow{H^+} (CH_3O)_2P(S)SCH_2COOH + CH_3NH_2$
28	$HSCH_2CONHCH_3 + H_2O \xrightarrow{H^+} HSCH_2COOH + CH_3NH_2$
29	$2HSCH_2COZ \xrightarrow{[O]} ZCOCH_2SSCH_2COZ$
30	$(CH_3O)_2P(S)SCH_2COOCH_3 + CH_3NH_2 \longrightarrow (CH_3O)_2P(S)SCH_2CONHCH_3$
31	$ClCH_2COOH + H_2O \xrightarrow{OH^-} HOCH_2COOH$
32	$HOCH_2COOH + ClCH_2COOH \xrightarrow{OH^-} HOOCCH_2OCH_2COOH$
33	$CH_3NCO + H_2O \longrightarrow CH_3NH_2 + CO_2$
34	$CH_3NCO + CH_3NH_2 \longrightarrow CH_3NHCONHCH_3$
35	$(CH_3O)_2P(S)SCH_2COOH + CH_3NCO \longrightarrow (CH_3O)_2P(S)SCH_2CONHCH_3 + CO_2$

(a) R and R' = alkyl
R'' = methyl
R''' = methyl or H
Y = oxygen or sulfur
Z = $-NHCH_3$, $-N(CH_3)_2$, $-OH$, or $-OCH_3$.

TABLE B-24. POSSIBLE CONTAMINANTS IN DIMETHOATE

Number	Contaminant	Precursor$^{(a)}$	Reaction Number$^{(b)}$	Step$^{(c)}$	Likelihood of Being Present in Final Product$^{(d)}$
1	P_2S_5	SM	—	1	0
2	CH_3OH	SM 27	— 20	1 3	+
3	$(CH_3O)_2P(S)SH$	1 + 2	1	1	++
4	$(CH_3O)_2(CH_3S)PS$	2 + 3	2	1	++
5	$(CH_3O)_3PS$	2 + 3 2 + 4	3 4	1	++
6	$(CH_3O)(CH_3S)_2PS$	1 + 4	5	1	+
7	$(CH_3O)(CH_3S)P(S)SH$	3 3	4 6	1	+
8	$(CH_3O)_2P(S)OH$	3 5	7 8	1	+
9	$(CH_3O)_2P(O)SH$	8	9	1	+
10	$(CH_3O)_2P(O)OH$	9	7	1	+
11	$(CH_3O)(CH_3S)P(O)SH$	6	8 + 9	1	0
12	$(CH_3S)_2P(S)SH$	3	6	1	0
13	$(CH_3O)_2P(S)SP(S)(OCH_3)_2$	3	10	1	+
14	$(CH_3O)_2P(S)SSP(S)(OCH_3)_2$	3	11	1	++

TABLE B-24. (Continued)

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
15	$(CH_3O)_2PSH$	3	12	1	0
16	$(CH_3O)_3P$	15	13	1	0
17	$(CH_3O)_2(CH_3S)P$	15	14	1	0
18	$(CH_3O)_2P(S)SP(OCH_3)_2$	15	15	1	0
19	$(CH_3O)_2(CH_3S)PO$	5	16	1	+
20	$(CH_3O)_3PO$	19 5	4 17	1	0
21	CH_3SH	4	4	1	0
22	$(CH_3S)_3PS$	6 + 21	4	1	0
23	$(CH_3S)_3PO$	22	17	1	0
24	$ClCH_2COOH$	SM	—	2	0
25	$Cl_2CHCOOH$	Im-24	—	—	0
26	Cl_3CCOOH	Im-24	—	—	0
27	$ClCH_2COOCH_3$	2 + 24	18	2	+
28	$Cl_2CHCOOCH_3$	2 + 25	18	2	+
29	$Cl_3CCOOCH_3$	2 + 26	18	2	0
30	CH_3NH_2	SM 40	— 27	3 St	+

TABLE B-24. (Continued)

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
31	$(CH_3)_2NH$	Im-30	—	—	0
32	$ClCH_2CONHCH_3$	27 + 30	19	3	++
33	$Cl_2CHCONHCH_3$	28 + 30	20	3	+
34	$CH_3NHCH_2CONHCH_3$	30 + 32	21	3	+
35	$ClCH_2CON(CH_3)_2$	27 + 31	19	3	+
36	$CH_3NHCH_2CO(NCH_3)_2$	30 + 35	21	3	0
37	$(CH_3)_2NCH_2CONHCH_3$	31 + 32	21	3	0
38	$(CH_3)_2NCH_2CON(CH_3)_2$	31 + 35	21	3	0
39	$CH_3N(CH_2CONHCH_3)_2$	32 + 34	22	3	0
40	$(CH_3O)_2P(S)SCH_2CONHCH_3$	3 + 32 30 + 43 42 + 55	23 30 35	4 A-4 B-3	Dimethoate
41	$(CH_3O)_2P(S)SCH_2CON(CH_3)_2$	3 + 35	23	4	++
42	$(CH_3O)_2P(S)SCH_2COOH$	3 + 24 40	23 27	4 or B-2 St	++
43	$(CH_3O)_2P(S)SCH_2COOCH_3$	3 + 27	23	4 or A-3	+
44	$(CH_3O)(CH_3S)P(O)SCH_2CONHCH_3$	40	16	4 or St	++
45	$(CH_3O)_2P(O)SCH_2CONHCH_3$	40	17	St	+

TABLE B-24. (Continued)

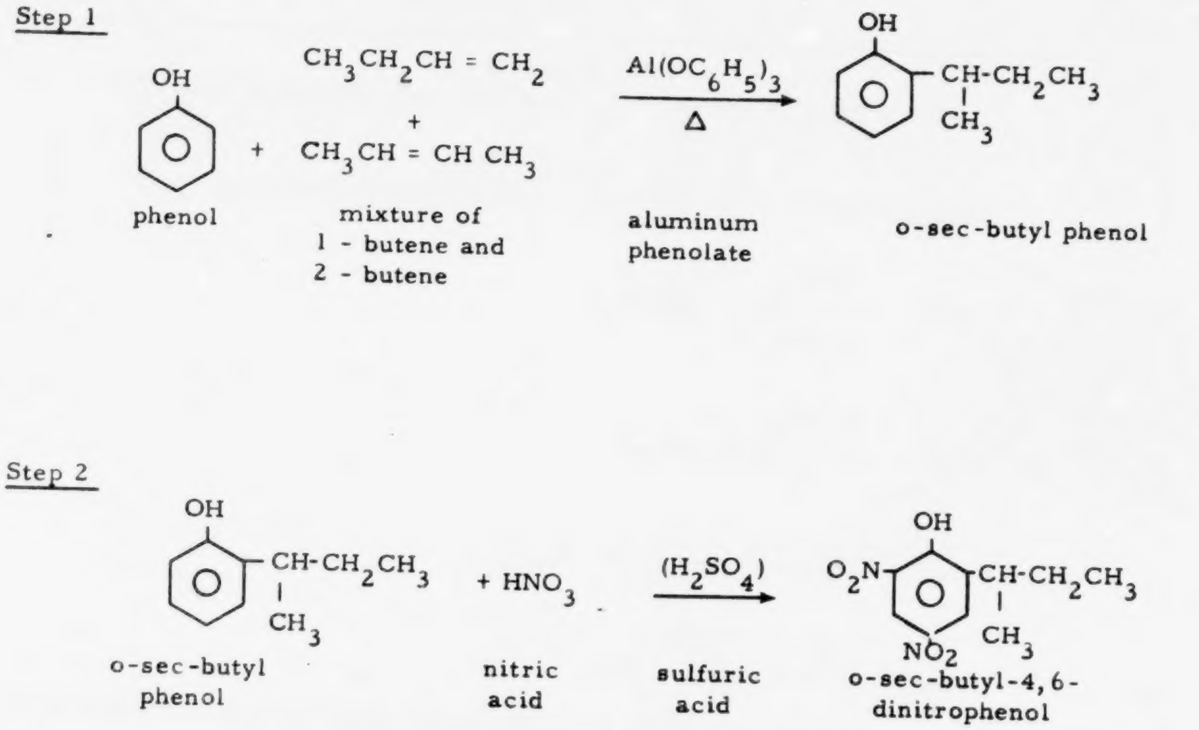
Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
46	$(CH_3O)(CH_3S)P(S)SCH_2CONHCH_3$	7 + 32	23	4	+
47	$(CH_3S)_2P(S)SCH_2CONHCH_3$	12 + 32	23	4	0
48	$[(CH_3O)_2P(S)S]_2CHCONHCH_3$	3 + 33	24	4	0
49	$CH_3OCH_2CONHCH_3$	2 + 32	25	4	0
50	$HSCH_2CONHCH_3$	40	26	St	+
51	$HSCH_2COOH$	50	28	St	+
52	$CH_3NHCOCH_2SSCH_2CONHCH_3$	50	29	St	+
53	$HOCH_2COOH$	24	31	B-2	0
54	$HOOCCH_2OCH_2COOH$	24 + 53	32	B-2	0
55	CH_3NCO	SM	—	B-3	0
56	$CH_3NHCONHCH_3$	55	33 + 34	B-3	+

(a) Precursor refers to the contaminant numbered in this table. SM = starting material; Im-24 = impurity in No. 24; Im-30 = impurity in No. 30.

(b) See Table B-23.

(c) The step (Figure B-13 or Figure B-14) during which the contaminant might be formed. St indicates possible formation during storage.

(d) 0 = likely to be present only at levels below 1 ppm
+ = likely to be present at levels above 1 ppm but below 100 ppm
++ = likely to be present at levels above 100 ppm.


Step 1
OH
phenol
+
CH3CH2CH = CH2
+
CH3CH = CH CH3
mixture of
1 - butene and
2 - butene
Al(OC6H5)3
Δ
aluminum
phenolate
OH
CH-CH2CH3
CH3
o-sec-butyl phenol
Step 2
OH
CH-CH2CH3
CH3
o-sec-butyl
phenol
+ HNO3
nitric
acid
(H2SO4)
sulfuric
acid
OH
O2N
CH-CH2CH3
CH3
NO2
o-sec-butyl-4, 6-
dinitrophenol

(Used in the form of various salts with ammonia or amines)

FIGURE B-15. SYNTHESIS OF DINOSEB

TABLE B-25. REACTIONS OCCURRING DURING PRODUCTION OF DINOSEB

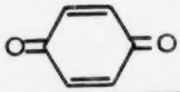
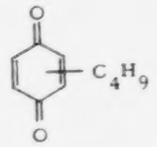
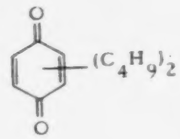
Reaction Number	Generalized Reaction
1	$\mathrm{ArOH + RR'C = CR''R''' \xrightarrow{Al(OC_6H_5)_3} Ar(OH)CRR'CHR''R'''}$
2	$\mathrm{ArOH + RR'C = CR''R''' \xrightarrow{Al(OC_6H_5)_3} ArOCRR'CHR''R'''}$
3	$\mathrm{ArH \xrightarrow[H_2SO_4]{HNO_2} ArNO_2}$
4.	$\mathrm{ArH \xrightarrow{H_2SO_4} ArSO_3H}$
5	$\mathrm{ArOH + ArSO_3H \longrightarrow ArSO_2Ar}$
6	$\mathrm{ArOH \xrightarrow{[O]}}$ 

TABLE B-26. POSSIBLE CONTAMINANTS IN DINOSEB

Number	Contaminant[a]	Precursor[b]	Reaction Number[c]	Step[d]	Likelihood of Being Present in Final Product[e]
1	C_6H_5OH	SM	–	1	o
2	C_4H_8	SM	–	1	o
3	$Al(OC_6H_5)_3$	Cat	–	1	o
4	$C_4H_9C_6H_4OH$	1+2	1	1	+
5	$(C_4H_9)_2C_6H_3OH$	4+2	1	1	+
6	$(C_4H_9)_3C_6H_2OH$	5+2	1	1	+
7	$C_6H_5OC_4H_9$	1+2	2	1	o
8	$C_4H_9C_6H_4OC_4H_9$	4+2	2	1	o
9	$C_4H_9C_6H_3(NO_2)OH$	4	3	2	++
10	$C_4H_9C_6H_2(NO_2)_2OH$	9	3	2	DINOSEB
11	$(C_4H_9)_2C_6H_2(NO_2)OH$	5	3	2	++
12	$C_6H_4(NO_2)OC_4H_9$	7	3	2	+
13	$C_6H_3(NO_2)_2OC_4H_9$	12	3	2	+
14	$C_4H_9C_6H_3(NO_2)OC_4H_9$	8	3	2	+
15	$C_4H_9C_6H_2(NO_2)_2OC_4H_9$	14	3	2	++
16	$C_6H_4(NO_2)OH$	1	3	2	+
17	$C_6H_3(NO_2)_2OH$	16	3	2	++
18	$C_6H_2(NO_2)_3OH$	17	3	2	+
19	$C_4H_9C_6H_3(SO_3H)OH$	4	4	2	+
20	$C_4H_9C_6H_2(SO_3H)(NO_2)OH$	19	3	2	++
21	$(C_4H_9)_2C_6H_2(SO_3H)OH$	5	4	2	+
22	$C_6H_4(SO_3H)OC_4H_9$	7	4	2	o
23	$C_6H_3(SO_3H)(NO_2)OC_4H_9$	22	3	2	+
24	$C_4H_9C_6H_3(SO_3H)OC_4H_9$	8	4	2	+
25	$C_4H_9C_6H_2(SO_3H)(NO_2)OC_4H_9$	24	3	2	+
26	$C_6H_4(SO_3H)OH$	1	4	2	o
27	$C_6H_3(SO_3H)(NO_2)OH$	26	3	2	+
28	$C_6H_2(SO_3H)(NO_2)_2OH$	27	3	2	o
29	$HOC_6H_3(C_4H_9)SO_2C_6H_3(C_4H_9)OH$	19+4	5	2	o

TABLE B-26. (Continued)

Number	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
30	$HOC_6H_2(C_4H_9)(NO_2)SO_2C_6H_3(C_4H_9)OH$	20+4	5	2	+
31	$HOC_6H_2(C_4H_9)(NO_2)SO_2C_6H_2(C_4H_9)(NO_2)OH$	20+9	5	2	o
32	$HOC_6H_2(C_4H_9)(NO_2)SO_2C_6H_2(C_4H_9)(NO_2)OC_4H_9$	20+14	5	2	o
33	$HOC_6H_2(C_4H_9)(NO_2)SO_2C_6H_3(NO_2)OH$	20+16	5	2	o
34	$HOC_6H_2(C_4H_9)_2SO_2C_6H_2(C_4H_9)(NO_2)OH$	21+9	5	2	o
35		4	6	2	+
36		5	6	2	o

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(a) There are a number of different positional isomers possible for most of the contaminants. The aromatic rings are substituted ortho and/or para to the -OH or -OC_4H_9. The C_4H_9 represents sec-butyl with traces of tert-butyl derived from isobutylene and traces of n-butyl from 1-addition instead of 2-addition.

(b) Precursor refers to the contaminant numbered in this table. SM = starting material. Cat = catalyst.

(c) **See Table B-25.**

(d) **The step (see Figure B-15) during which the contaminant might be formed**

(e) o = Likely to be present at levels below 1 ppm
+ = Likely to be present at levels above 1 ppm but below 100 ppm
++ = Likely to be present at levels above 100 ppm.

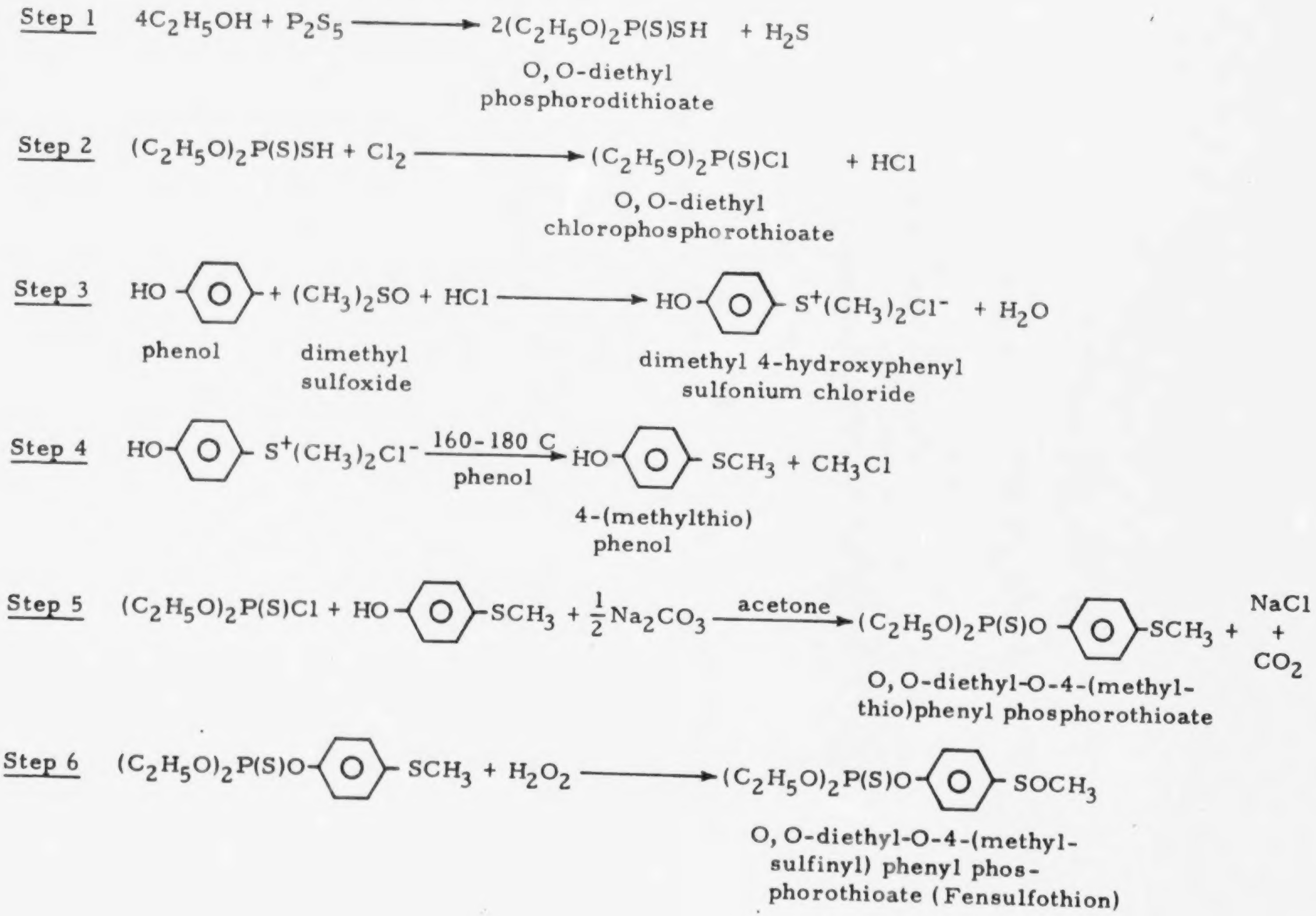

Step 1
4C2H5OH + P2S5
2(C2H5O)2P(S)SH + H2S
O, O-diethyl phosphorodithioate
Step 2
(C2H5O)2P(S)SH + Cl2
(C2H5O)2P(S)Cl + HCl
O, O-diethyl chlorophosphorothioate
Step 3
HO + (CH3)2SO + HCl
HO S+(CH3)2Cl- + H2O
phenol
dimethyl sulfoxide
dimethyl 4-hydroxyphenyl sulfonium chloride
Step 4
HO S+(CH3)2Cl-
160-180 C
phenol
HO SCH3 + CH3Cl
4-(methylthio) phenol
Step 5
(C2H5O)2P(S)Cl + HO SCH3 + 1/2 Na2CO3
acetone
(C2H5O)2P(S)O SCH3 + NaCl + CO2
O, O-diethyl-O-4-(methylthio)phenyl phosphorothioate
Step 6
(C2H5O)2P(S)O SCH3 + H2O2
(C2H5O)2P(S)O SOCH3
O, O-diethyl-O-4-(methylsulfinyl) phenyl phosphorothioate (Fensulfothion)

FIGURE B-16. SYNTHESIS OF FENSULFOTHION

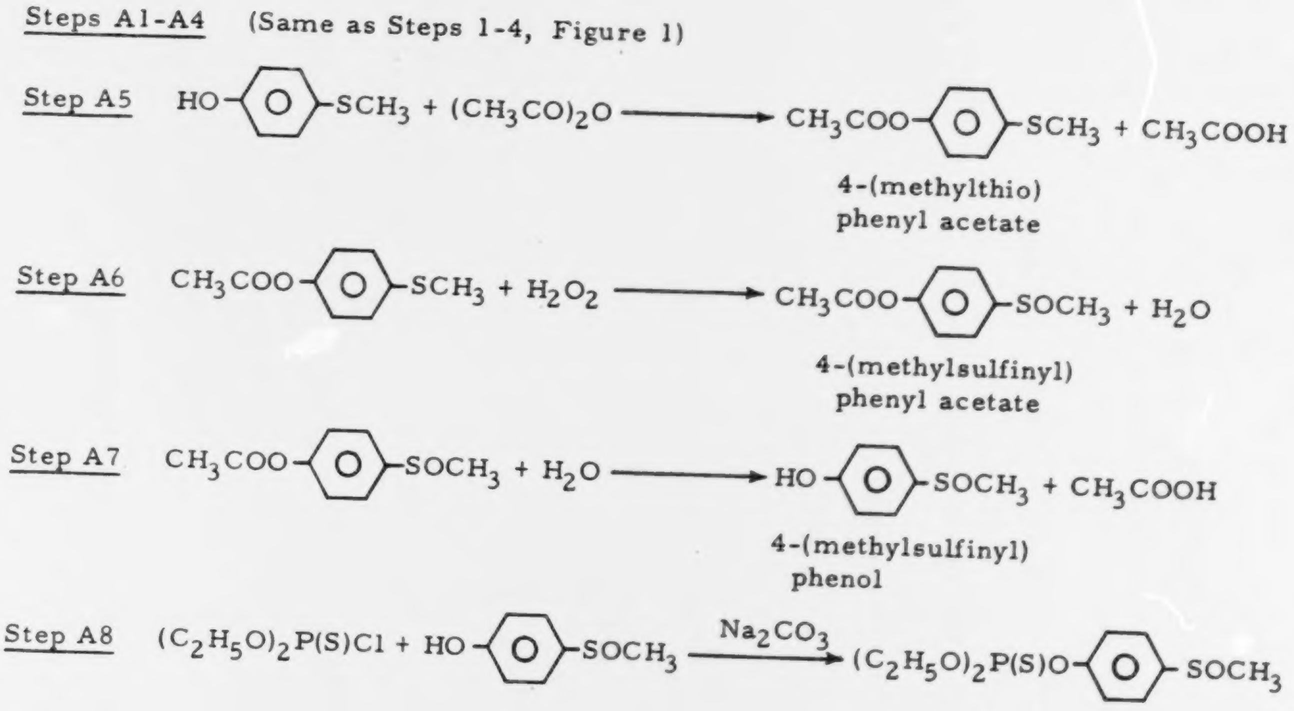
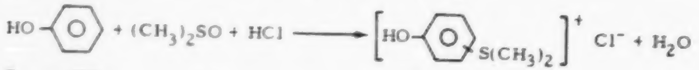
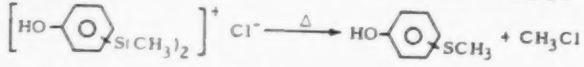
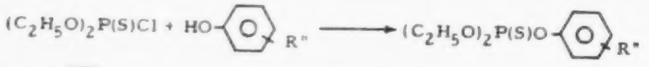
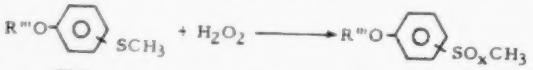
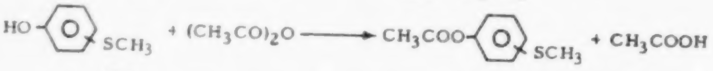
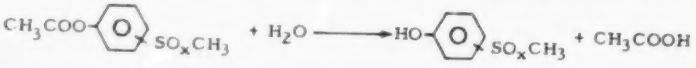
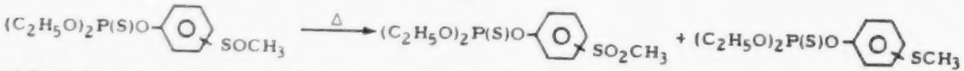

Steps A1-A4 (Same as Steps 1-4, Figure 1)
Step A5
HO-
-SCH3 + (CH3CO)2O
CH3COO-
-SCH3 + CH3COOH
4-(methylthio) phenyl acetate
Step A6
CH3COO-
-SCH3 + H2O2
CH3COO-
-SOCH3 + H2O
4-(methylsulfinyl) phenyl acetate
Step A7
CH3COO-
-SOCH3 + H2O
HO-
-SOCH3 + CH3COOH
4-(methylsulfinyl) phenol
Step A8
(C2H5O)2P(S)Cl + HO-
-SOCH3
Na2CO3
(C2H5O)2P(S)O-
-SOCH3

FIGURE B-17. ALTERNATE SYNTHESIS OF FENSULFOTHION

TABLE B-27. REACTIONS OCCURRING DURING PRODUCTION OF FENSULFOTHION

Number	Generalized Reaction[(a)]
1	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6	$(RO)_2P(Y)SH \xrightarrow{P_2S_5} (RO)(RS)P(Y)SH + (RS)_2P(Y)SH$
7	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
8	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$
9	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
10	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
11	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
12	$2(RY)_2P(Y)SH + Cl_2 \longrightarrow (RY)_2P(Y)SSP(Y)(RY)_2 + 2HCl$
13	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
14	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
15	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
16	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(RY)_2$
17	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
18	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
19	$(RY)_2P(S)SH + Cl_2 \longrightarrow (RY)_2P(S)Cl + S_2Cl_2$
20	 HO (CH3)2SO HCl HO S(CH3)2 Cl⁻ H2O
21	 HO S(CH3)2 Cl⁻ Δ HO SCH3 CH3Cl
22	 (C2H5O)2P(S)Cl HO R" (C2H5O)2P(S)O R"
23	 R'''O SCH3 H2O2 R'''O SOxCH3
24	 HO SCH3 (CH3CO)2O CH3COO SCH3 CH3COOH
25	 CH3COO SOxCH3 H2O HO SOxCH3 CH3COOH
26	 (C2H5O)2P(S)O SOCH3 Δ (C2H5O)2P(S)O SO2CH3 (C2H5O)2P(S)O SCH3
27	$(RO)_2P(S)OH + H_2O \longrightarrow ROP(S)(OH)_2 + ROH$
28	$ROP(S)(OH)_2 + 2H_2O \longrightarrow ROH + H_3PO_4 + H_2S$

(a) R and R' = alkyl, aryl or substituted aryl
R" = H, CH_3S-, CH_3SO- or CH_3SO_2.
R''' = CH_3CO-, $(C_2H_5O)_2P(S)$- or $(C_2H_5O)(C_2H_5S)P(O)$-
Y = oxygen or sulfur.
x = 1 or 2.

TABLE B-28. POSSIBLE CONTAMINANTS IN PENSULFOTHION

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Probability of Being Present in Final Product(d)
1	P_2S_5	SM	--	1	0
2	C_2H_5OH	SM 38	-- 8 + 27	1 St	+
3	$(C_2H_5O)_2P(S)SH$	1 + 2	1	1	+
4	$(C_2H_5O)_2(C_2H_5S)PS$	2 + 3	2	1	++
5	$(C_2H_5O)_3PS$	2 + 3 2 + 4	3 4	1 1	++
6	$(C_2H_5O)(C_2H_5S)_2PS$	1 + 4	5	1	+
7	$(C_2H_5O)(C_2H_5S)P(S)SH$	3	6	1	+
8	$(C_2H_5O)_2P(S)OH$	3 5 38	7 8 8	1 1 St	+
9	$(C_2H_5O)_2P(O)SH$	8	9	1	0
10	$(C_2H_5O)_2P(O)OH$	9	7	1	0
11	$(C_2H_5O)(C_2H_5S)P(O)SH$	6	8 + 9	1	0
12	$(C_2H_5S)_2P(S)SH$	3	6	1	0
13	$(C_2H_5O)_2P(S)SP(S)(OC_2H_5)_2$	3	10	1	+
14	$(C_2H_5O)_2P(S)SSP(S)(OC_2H_5)_2$	3 3	11 12	1 2	++
15	$(C_2H_5O)_2PSH$	3	13	1	0
16	$(C_2H_5O)_3P$	15	14	1	0
17	$(C_2H_5O)_2(C_2H_5S)P$	15	15	1	0
18	$(C_2H_5O)_2P(S)P(OC_2H_5)_2$	15	16	1	0
19	$(C_2H_5O)_2(C_2H_5S)PO$	5	17	1	+
20	$(C_2H_5O)_3PO$	2 + 19	4	1	0
21	C_2H_5SH	2 + 4	4	1	0
22	$(C_2H_5S)_3PS$	6 + 21	4	1	0
23	$(C_2H_5S)_3PO$	22	18	1	0
24	Cl_2	SM	--	2	0
25	$(C_2H_5O)_2P(S)Cl$	3 + 24	19	2	++
26	C_6H_5OH	SM	--	3	++
27	$(CH_3)_2SO$	SM	--	3	+

TABLE B-28. (Continued)

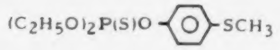
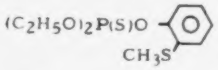
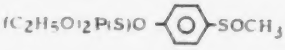
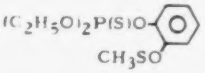
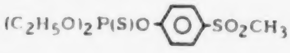
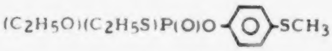
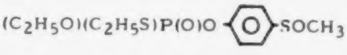
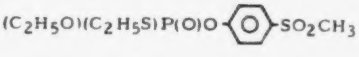
Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Probability of Being Present in Final Product[d]
28	$[4\text{-}HOC_6H_4S(CH_3)_2]^+ Cl^-$	26 + 27	20	3	+
29	$[2\text{-}HOC_6H_4S(CH_3)_2]^+ Cl^-$	26 + 27	20	3	0
30	$4\text{-}HOC_6H_4SCH_3$	28	21	4	++
31	$2\text{-}HOC_6H_4SCH_3$	29	21	4	+
32	CH_3Cl	28	21	4	0
33	CH_3COCH_3	Solv.	--	5	0
34	 (C2H5O)2P(S)O SCH3	25 + 30 38	22 26	5 St	++
35	 (C2H5O)2P(S)O CH3S	25 + 31	22	5	+
36	H_2O_2	SM	--	6 A6	0
37	CH_3COOH	Solv.		6	0
38	 (C2H5O)2P(S)O SOCH3	34 + 36 25 + 45	23 22	6 A8	Fensulfothion
39	 (C2H5O)2P(S)O CH3SO	35 + 36 25 + 46	23 22	6 A8	++
40	$(CH_3CO)_2O$	SM	--	A5	0
41	$4\text{-}CH_3COOC_6H_4SCH_3$	30 + 40	24	A5	0
42	$2\text{-}CH_3COOC_6H_4SCH_3$	31 + 40	24	A5	0
43	$4\text{-}CH_3COOC_6H_4SOCH_3$	36 + 41	23	A6	0
44	$2\text{-}CH_3COOC_6H_4SOCH_3$	36 + 42	23	A6	0
45	$4\text{-}HOC_6H_4SOCH_3$	43	25	A7	++
46	$2\text{-}HOC_6H_4SOCH_3$	44	25	A7	+
47	$4\text{-}CH_3COOC_6H_4SO_2CH_3$	36 + 41	23	A6	0
48	$4\text{-}HOC_6H_4SO_2CH_3$	47	25	A7	+
49	 (C2H5O)2P(S)O SO2CH3	25 + 48 38	22 26	A8 St	++
50	 (C2H5O)(C2H5S)P(O)O SCH3	34	17	5	+
51	 (C2H5O)(C2H5S)P(O)O SOCH3	38	17	6 or St	++
52	 (C2H5O)(C2H5S)P(O)O SO2CH3	49 50	17 23	6 or St 6	+

TABLE B-28. (Continued)

Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Probability of Being Present in Final Product[d]
53	$C_2H_5OPS(OH)_2$	8	27	St	+
54	H_3PO_4	53	28	St	+
55	H_2S	53	28	St	+
56	$(C_2H_5O)_2P(S)OC_6H_5$	25 + 26	22	5	++
57	$(C_2H_5O)(C_2H_5S)P(O)OC_6H_5$	56	17	5	++

(a) Precursor refers to the contaminant numbered in this table. SM = starting material; Solv. = solvent.

(b) See Table B-27.

(c) The step (Figure B-16 or Figure B-17) during which the contaminant might be formed. ısible St indicates possible formation during storage

(d) o probably present only at levels below 1 ppm.
+ probably present at levels above 1 ppm but below 100 ppm.
++ probably present at levels above 100 ppm.

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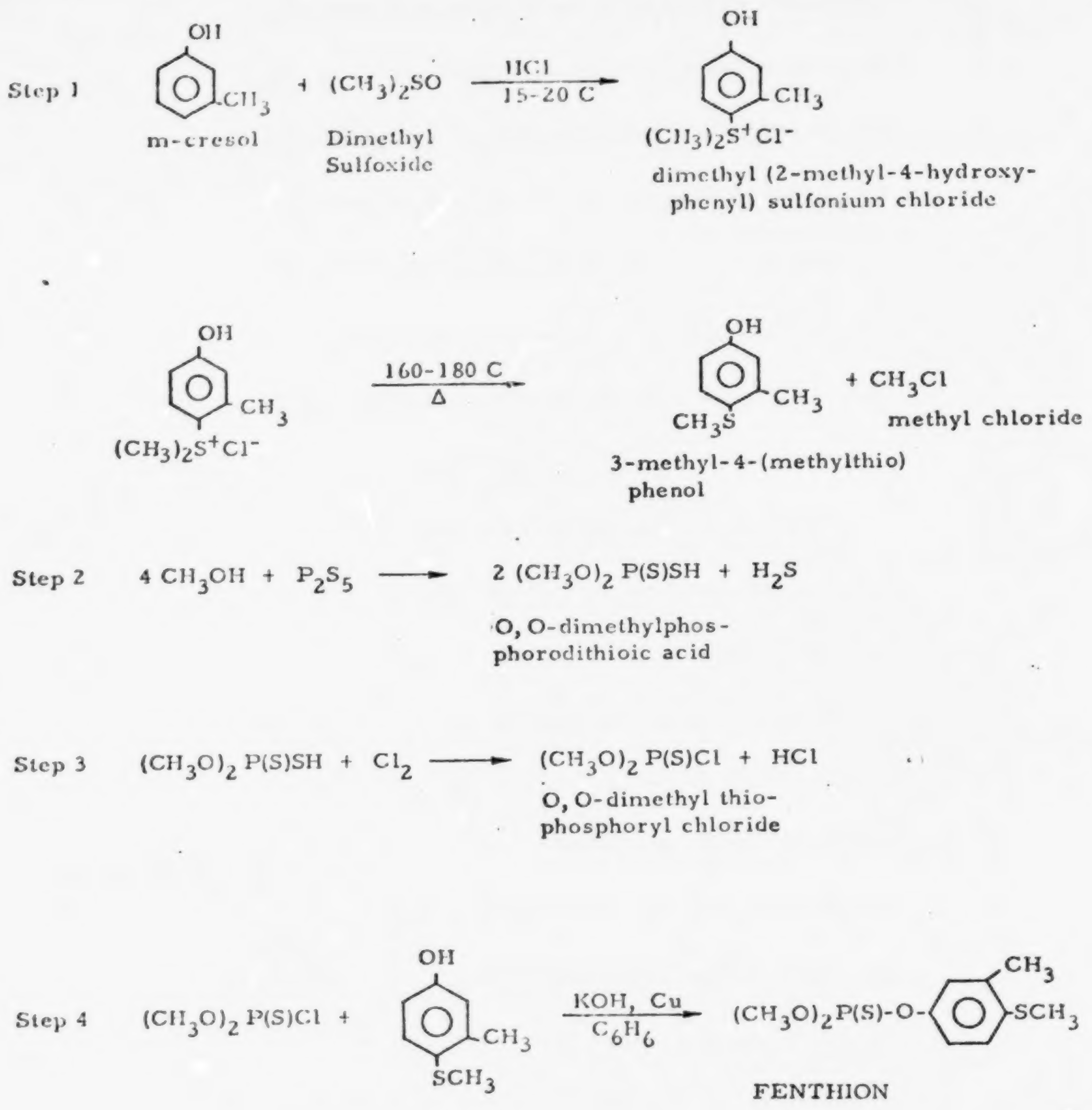

Step 1
OH
CH3
+ (CH3)2SO
HCl
15-20 C
OH
CH3
(CH3)2S+ Cl-
m-cresol
Dimethyl Sulfoxide
dimethyl (2-methyl-4-hydroxy-phenyl) sulfonium chloride
OH
CH3
(CH3)2S+Cl-
160-180 C
Δ
OH
CH3
CH3S
+ CH3Cl
methyl chloride
3-methyl-4-(methylthio) phenol
Step 2
4 CH3OH + P2S5
2 (CH3O)2 P(S)SH + H2S
O, O-dimethylphosphorodithioic acid
Step 3
(CH3O)2 P(S)SH + Cl2
(CH3O)2 P(S)Cl + HCl
O, O-dimethyl thiophosphoryl chloride
Step 4
(CH3O)2 P(S)Cl +
OH
CH3
SCH3
KOH, Cu
C6H6
(CH3O)2P(S)-O-
CH3
SCH3
FENTHION

FIGURE B-18. SYNTHESIS OF FENTHION

TABLE B-29. REACTIONS OCCURRING DURING PRODUCTION ON FENTHION

Reaction Number	Generalized Reaction[a]
1	$C_6H_4(OH)(CH_3) + (CH_3)_2SO \longrightarrow C_6H_3(OH)(CH_3)[{}^+S(CH_3)_2\,Cl^-]$
2	$C_6H_3(OH)(CH_3)[\overset{+}{S}(CH_3)_2\,Cl^-] \longrightarrow C_6H_3(OH)(CH_3(SCH_3) + CH_3Cl$
3	$C_6H_3(OH)(CH_3)_2 + (CH_3)_2SO \longrightarrow C_6H_2(OH)_2(CH_3)_2[\overset{+}{S}(CH_3)_2\,Cl^-]$
4	$C_6H_2(OH)(CH_3)_2[\overset{+}{S}(CH_3)_2\,Cl^-] \longrightarrow C_6H_3(OH)(CH_3)_2(SCH_3)$
5	$C_6H_4(OH)(CH_3) + 2(CH_3)_2SO \longrightarrow C_6H_2(OH)(CH_3)[\overset{+}{S}(CH_3)_2\,Cl^-]_2$
6	$C_6H_3(OH)(CH_3)[\overset{+}{S}(CH_3)_2\,Cl^-]_2 \longrightarrow C_6H_2(OH)(H_3)(SCH_3)_2$
7	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S + ROP(S)(SH)_2 + RO_2PS$
8	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
9	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
10	$(RY)_2P(Y)SR + R'OH \longrightarrow (RY)_2P(Y)OR' + RSH$
11	$2RSH \xrightarrow{[O]} RSSR$
12	$(RY)_2P(Y)OR + P_2S_5 \longrightarrow (RY)_2P(Y)SR$
13	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
14	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
15	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)\ Y\ P(Y)(RY)_2 + H_2Y$
16	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SS\ P(Y)(RY)_2$
17	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
18	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
19	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
20	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$

TABLE B-29. (Continued)

Reaction Number	Generalized Reaction[(a)]
21	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(RY)_2$
22	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
23	$(RY)_2P(Y)SR + H_2O \longrightarrow (RY)_2P(Y)SH + ROH$
24	$(RY)_2P(Y)SR + H_2O \longrightarrow (RY)_2P(Y)OH + RSH$
25	$(RY)_2P(Y)OR + H_2O \longrightarrow (RY)_2P(Y)OH + ROH$
26	$(RY)_2P(S)SH + Cl_2 \longrightarrow (RY)_2P(S)Cl + S_2Cl_2$
27	$2(RY)_2P(S)SH + Cl_2 \longrightarrow (RY)_2P(S)SSP(S)(YR)_2 + HCl$
28	$(RY)_2P(Y)SH + Cl_2 \longrightarrow (RY)_2P(Y)Cl + S_2Cl_2$
29	$(RY)_2P(S)OH + Cl_2 \longrightarrow (RY)_2P(S)Cl + HCl$
30	$(RY)_2P(O)SH + Cl_2 \longrightarrow (RY)_2P(O)Cl + S_2Cl_2$
31	$(RY)_2PSH + Cl_2 \longrightarrow (RY)_2PCl + S_2Cl_2$
32	$(RY)_2P(Y)OH + Cl_2 \longrightarrow (RY)_2P(Y)Cl + HCl$
33	$(RY)P(S)(SH)_2 + Cl_2 \longrightarrow RY\,P(S)Cl_2 + S_2Cl_2$
34	$(RY)P(O)(SH)_2 + Cl_2 \longrightarrow RYP(O)Cl_2 + S_2Cl_2$
35	$ROH + S_2Cl_2 \longrightarrow ROSSR + HCl$
36	$(RY)_2P(S)Cl + R''OH \longrightarrow (RY)_2P(S)(OR'') + NaCl$
37	$(RY)_2P(Y)Cl + R''OH \longrightarrow (RY)_2P(Y)OR'' + NaCl$
38	$(RY)_2P(O)Cl + R''OH \longrightarrow (RY)_2P(O)OR'' + NaCl$
39	$(RY)_2PCl + R''OH \longrightarrow (RY)_2POR'' + NaCl$
40	$(RY)P(S)Cl_2 + 2R''OH \longrightarrow RYP(S)(OR'')_2 + NaCl$

TABLE B-29. (Continued)

Reaction Number	Generalized Reaction[(a)]
41	$(RY)P(O)Cl_2 + R''OH \longrightarrow RY\,P(O)(OR'')_2 + NaCl$
42	$S_2Cl_2 + 2R''OH \longrightarrow R''\text{-}O\text{-}S\text{-}S\text{-}O\text{-}R'' + NaCl$
43	$(RY)_2P(S)OR'' \rightleftharpoons (RY)_2P(O)SR''$
44	$RY\,P(S)(OR'')_2 \rightleftharpoons RY\,P(O)(SR'')(OR'')$
45	$(RY)_2P(Y)OR \rightleftharpoons (RY)_2P(O)YR''$
46	$(RY)_2P(S)OR'' + H_2O \longrightarrow (RY)_2P(S)OH + R''OH$
47	$RY\,P(S)(OR'')_2 + H_2O \longrightarrow RY\,P(S)(OH)_2 + ROH$
48	$(RY)_2(P)(O)(SR'') + H_2O \longrightarrow (RY)_2P(O)OH + R''SH$
49	$(RY)_2P(Y)OR'' + H_2O \longrightarrow (RY)_2P(Y)OH + R''OH$
50	$(RY)_2P(O)(SR'')(OR'') + H_2O \longrightarrow (RY)_2P(O)(OH)_2 + \ddot{R}SH + R''OH$
51	$(RY)_2POR'' + H_2O \longrightarrow (RY)_2POH + R''OH$
52	$R''OH + S_2Cl_2 \longrightarrow R''OSSOR'' + NaCl$
53	$R''SH + S_2Cl_2 \longrightarrow RSSSSR'' + NaCl$
54	$\ddot{R}OSSOR'' + H_2O \longrightarrow 2R''OH + S_2$
55	$R''SSSSR'' + H_2O \longrightarrow 2RSH + S_2$
56	$R''SH \xrightarrow{[O]} R''SSR''$
57	$(RY)_2P(S)OC_6H_4(SCH_3)(CH_3) \xrightarrow{[O]} (RY)_2P(S)OC_6H_4(SOCH_3)(CH_3)$
58	$(RY)_2P(S)OC_6H_4(SCH_3)(CH_3) \xrightarrow{[O]} (RY)_2P(S)OC_6H_4(SO_2CH_3)(CH_3)$
59	$(RY)_2P(S)OC_6H_4SOCH_3 + H_2O \longrightarrow (RY)_2P(S)OH + HOC_6H_4(SOCH_3)(CH_3)$
60	$(RY)_2P(S)OC_6H_4SO_2CH_3 + H_2O \longrightarrow (RY)_2P(S)OH + HOC_6H_4(SO_2CH_3)(CH_3)$

TABLE B-29. (Continued)

Reaction Number	Generalized Reaction[(a)]
61	$\mathrm{(RY)_2P(O)SC_6H_4SCH_3 \xrightarrow{[O]} (RY)_2P(O)SC_6H_3(SOCH_3)(CH_3)}$
62	$\mathrm{(RY)_2P(O)SC_6H_4SCH_3 \xrightarrow{[O]} (RY)_2P(O)SC_6H_3(SO_2CH_3)(CH_3)}$
63	$\mathrm{(RY)_2P(O)SC_6H_4(SOCH_3)(CH_3) + H_2O \longrightarrow (RY)_2P(O)OH + HSC_6H_3(SOCH_3)(CH_3)}$
64	$\mathrm{(RY)_2P(O)SC_6H_4(SO_2CH_3)(CH_3) + H_2O \longrightarrow (RY)_2P(O)OH + HSC_6H_3(SO_2CH_3)(CH_3)}$
65	$\mathrm{(RY)_2P(O)OC_6H_4(SCH_3)(CH_3) \xrightarrow{[O]} (RY)_2P(O)OC_6H_4(SOCH_3)(CH_3)}$
66	$\mathrm{(RY)_2P(O)OC_6H_4(SCH_3)(CH_3) \xrightarrow{[O]} (RY)_2P(O)OC_6H_4(SO_2CH_3)(CH_3)}$
67	$\mathrm{(RY)_2P(O)OC_6H_4(SOCH_3)(CH_3) + H_2O \longrightarrow (RY)_2P(O)OH + HOC_6H_4(SOCH_3)(CH_3)}$
68	$\mathrm{(RY)_2P(O)OC_6H_4(SO_2CH_3)(CH_3) + H_2O \longrightarrow (RY)_2P(O)OH + HOC_6H_4(SO_2CH_3)(CH_3)}$
69	$\mathrm{RY\ P(S)(OC_6H_4(SCH_3)(CH_3) + H_2O \longrightarrow RY\ P(S)OH + HOC_6H_4(CH_3)(SCH_3)}$
70	$\mathrm{(CH_3S)(CH_3)C_6H_3YH \xrightarrow{[O]} (CH_3SO)(CH_3)C_6H_3YH}$
71	$\mathrm{(CH_3S)(CH_3)C_6H_3YH \xrightarrow{[O]} (CH_3SO_2)(CH_3)C_6H_3YH}$
72	$\mathrm{[(CH_3S)(CH_3)C_6H_3S\text{-}]_2 \xrightarrow{[O]} [(CH_3SO)(CH_3)(C_6H_3S\text{-})]_2 + [(CH_3SO_2)(CH_3)C_6H_3S\text{-}]_2}$

(a) R and R' = alkyl or substituted alkyl
Y = O or S
R'' = 3-methyl-4-methylthiophenyl.

TABLE B-30. POSSIBLE CONTAMINANTS IN FENTHION

No.	Contaminant[a]	Precursor[b]	Reaction Number[c]	Step[d]	Likelihood of Being Present in Final Product[e]
1	$C_6H_4(CH_3)(OH)$	SM	1	1	0
2	CH_3SOCH_3	SM	1	1	0
3	$C_6H_3(CH_3)(OH)[S(CH_3)_2^+ Cl^-]$	1+2	1	1	0
4	$C_6H_2(CH_3)(OH)[S(CH_3)_2^+Cl^-]_2$	1+2	5	1	0
5	$C_6H_3(CH_3)_2(OH)$	SM	3	1	0
6	$C_6H_2(CH_3)_2(OH)[S(CH_3)_2^+Cl^-]$	5+2	3	1	0
7	$C_6H(CH_3)_2(OH)[S(CH_3)_2^+Cl^-]_2$	5+2	3	1	0
8	$C_6H_3(CH_3)(OH)(SCH_3)$	3	2	1	+
9	$C_6H_2(CH_3)(OH)(SCH_3)_2$	4	6	1	0
10	$C_6H_2(CH_3)_2(OH)(SCH_3)$	6	4	1	0
11	$C_6H(CH_3)_2(OH)(SCH_3)_2$	7	4	1	0
12	MeOH	SM	7	2	0
13	P_2S_5	SM	7	2	0
14	$(MeO)_2P(S)SH$	12+13	7	2	+
15	$MeOP(S)(SH)_2$	12+13	7	2	+
16	$(MeO)_3PS$	12+13	7	2	++
17	$(MeO)_2(MeS)PS$	14+13	12	2	0
18	$(MeO)(MeS)_2PS$	17	12	2	0
19	$(MeO)(MeS)P(S)SH$	14	23	2	0

TABLE B-30. (Continued)

No.	Contaminant[a]	Precursor[b]	Reaction Number[c]	Step[d]	Likelihood of Being Present in Final Product[e]
20	$(MeO)_2P(S)OH$	14	24	2	0
21	$(MeO)_2P(O)SH$	20	14	2	0
22	$(MeO)_2P(O)OH$	21	24	2	0
23	$(MeO)(MeS)P(O)OH$	19	13	2	0
24	$(MeS)_2P(S)SH$	19	12	2	0
25	$(MeO)_2P(S)SP(S)(OMe)_2$	14	15	2	0
26	$(MeO)_2P(S)SSP(S)(OMe)_2$	14	16	2	0
27	$(MeO)_2PSH$	14	18	2	0
28	$(MeO)_3P$	26	22	2	0
29	$(MeO)_2(MeS)PO$	26	22	2	0
30	$(MeO)_3PO$	16	22	2	0
31	$(MeS)_3PS$	16	7	2	0
32	$(MeS)PO$	30	22	2	0
33	$(MeO)_2P(S)SP(OMe)_2$	26	21	2	0
34	$(MeO)_2(MeS)PO$	16	22	2	0
35	$(MeO)(MeS)_2PO$	18	22	2	0
36	$(MeO)_2(MeS)PO$	17	22	2	0
37	MeSH	35	10	2	0
38	MeSSMe	37	11	2	0

TABLE B-30. (Continued)

No.	Contaminant(a)	Precursor(b)	Reaction Number(c)	Step(d)	Likelihood of Being Present in Final Product(e)
39	$(MeO)_2P(S)Cl$	14+20	26, 32	3	+
40	$(MeO)P(S)Cl_2$	15	33	3	0
41	$(MeO)(MeS)P(S)Cl$	19	28	3	0
42	$(MeO)_2P(O)Cl$	21+22	30	3	0
43	$(MeO)(MeS)P(O)Cl$	22	30	3	0
44	$(MeS)_2P(S)Cl$	23	26	3	0
45	$(MeO)PCl$	26	31	3	0
46	$(CH_3S)(CH_3)C_6H_3OP(S)(OMe)_2$	38+8	36	4	Fenthion
47	$(CH_3S)_2(CH_3)C_6H_2OP(S)(OMe)_2$	38+9	36	4	+
48	$(CH_3S)(CH_3)_2C_6H_2OP(S)(OMe)_2$	38+10	36	4	0
49	$(CH_3S)_2(CH_3)_2C_6HOP(S)(OMe)_2$	38+11	36	4	0
50	$[(CH_3S)(CH_3)C_6H_3O]_2P(S)(OMe)$	39+8	40	4	++
51	$[(CH_3S)_2(CH_3)C_6H_2O]_2P(S)(OMe)$	39+9	40	4	0
52	$[(CH_3S)(CH_3)_2C_6H_2O]_2P(S)(OMe)$	39+10	40	4	0
53	$[(CH_3S)_2(CH_3)_2C_6HO]_2P(S)(OMe)$	39+11	40	4	0
54	$(CH_3S)(CH_3)C_6H_3O\ P(S)(OMe)(SMe)$	40+8	37	4	+
55	$(CH_3S)_2(CH_3)C_6H_2O\ P(S)(OMe)(SMe)$	40+9	37	4	0
56	$(CH_3S)(CH_3)_2C_6H_2O\ P(S)(OMe)(SMe)$	40+10	37	4	0
57	$(CH_3S)_2(CH_3)_2C_6HO\ P(S)(OMe)(SMe)$	40+11	37	4	0

TABLE B-30. (Continued)

No.	Contaminant(a)	Precursor(b)	Reaction Number(c)	Step(d)	Likelihood of Being Present in Final Product(e)
58	$(CH_3S)(CH_3)C_6H_3O\ P(O)(OMe)_2$	41+8	38	4	++
59	$(CH_3S)_2(CH_3)C_6H_2O\ P(O)(OMe)_2$	41+9	38	4	0
60	$(CH_3S)(CH_3)_2C_6H_2O\ P(O)(OMe)_2$	41+10	38	4	0
61	$(CH_3S)_2(CH_3)_2C_6HO\ P(O)(OMe)_2$	41+11	38	4	0
62	$(CH_3S)(CH_3)C_6H_3O\ P(O)(OMe)(SMe)$	42+8	38	4	+
63	$(CH_3S)_2(CH_3)C_6H_2O\ P(O)(OMe)(SMe)$	42+9	38	4	0
64	$(CH_3S)(CH_3)_2C_6H_2O\ P(O)(OMe)(SMe)$	42+10	38	4	0
65	$(CH_3S)_2(CH_3)_2C_6HO\ P(O)(OMe)(SMe)$	42+11	38	4	
66	$(CH_3S)(CH_3)C_6H_3O\ P(S)(SMe)_2$	43+8	37	4	+
67	$(CH_3S)_2(CH_3)C_6H_2O\ P(S)(SMe)_2$	43+9	37	4	0
68	$(CH_3S)(CH_3)_2C_6H_2O\ P(S)(SMe)_2$	43+10	37	4	0
69	$(CH_3S)_2(CH_3)_2C_6HO\ P(S)(SMe)_2$	43+11	37	4	0
70	$(CH_3S)(CH_3)C_6H_3O\ P(OMe)_2$	44+8	39	4	0
71	$(CH_3S)_2(CH_3)C_6H_2O\ P(OMe)_2$	44+9	39	4	0
72	$(CH_3S)(CH_3)_2C_6H_2O\ P(OMe)_2$	44+10	39	4	0
73	$(CH_3S)_2(CH_3)_2C_6HO\ P(OMe)_2$	44+11	39	4	0
74	$(CH_3S)(CH_3)C_6H_3SP(O)(OMe)_2$	45	43	4	+
75	$(CH_3S)_2(CH_3)C_6H_2S\ P(O)(OMe)_2$	46	43	4	0
76	$(CH_3S)(CH_3)_2C_6H_2S\ P(O)(OMe)_2$	47	43	4	0

TABLE B-30. (Continued)

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
77	$(CH_3S)_2(CH_3)_2C_6H_2S\ P(O)(OMe)_2$	48	43	4	0
78	$[(CH_3S)(CH_3)C_6H_3S][(CH_3S)(CH_3)C_6H_3O]\ P(O)(OMe)_2$	49	44	4	0
79	$(CH_3S)(CH_3)C_6H_3SP(O)(OMe)(SMe)$	53	43	4	+
80	$(CH_3S)(CH_3)C_6H_3S\ P(O)(SMe)_2$	65	43	4	+
81	$(CH_3S)(CH_3)C_6H_3SH$	77, 78, 79	48	4	++
82	$[(CH_3S)(CH_3)C_6H_3]_2S_2$	80	56	4	+
83	$(CH_3SO)(CH_3)C_6H_3SH$	80	70	4	+
84	$(CH_3SO_2)(CH_3)C_6H_3SH$	82	71	4	+
85	$[(CH_3SO)(CH_3)C_6H_3]_2S_2$	81	72	4	+
86	$[(CH_3SO_2)(CH_3)C_6H_3]_2S_2$	84	72	4	+
87	$(CH_3SO)(CH_3)C_6H_3O\ P(S)(OMe)_2$	45	57	4	++
88	$(CH_3SO$ [illegible] $C_6H_3O\ P(S)(OMe)_2$	86	58	4	++
89	$(CH_3S$ [illegible] $H_3S\ P(O)(OMe)_2$	73	61	4	++
90	$(CH_3SO_2)(CH_3)C_6H_3S\ P(O)(OMe)_2$	88	62	4	++
91	$(CH_3SO)(CH_3)C_6H_3O\ P(O)(OMe)_2$	57	65	4	++
92	$(CH_3SO_2)(CH_3)C_6H_3O\ P(O)(OMe)_2$	90	66	4	++
93	$(CH_3SO)(CH_3)C_6H_3OH$	86	59		++
94	$(CH_3SO_2)(CH_3)C_6H_3OH$	87	60	4	+

(a) There are a number of different positional isomers possible for some of the contaminants.
(b) Precursor refers to contaminant numbered in this table. SM = starting material.
(c) **See Table B-29.**
(d) **The step (Figure B-18) during which the contaminant might be formed.**
(e) 0 = likely to be present below 1 ppm.
+ = likely to be present above 1 ppm but below 100 ppm.
(f) ++ = likely to be present at levels above 100 ppm.

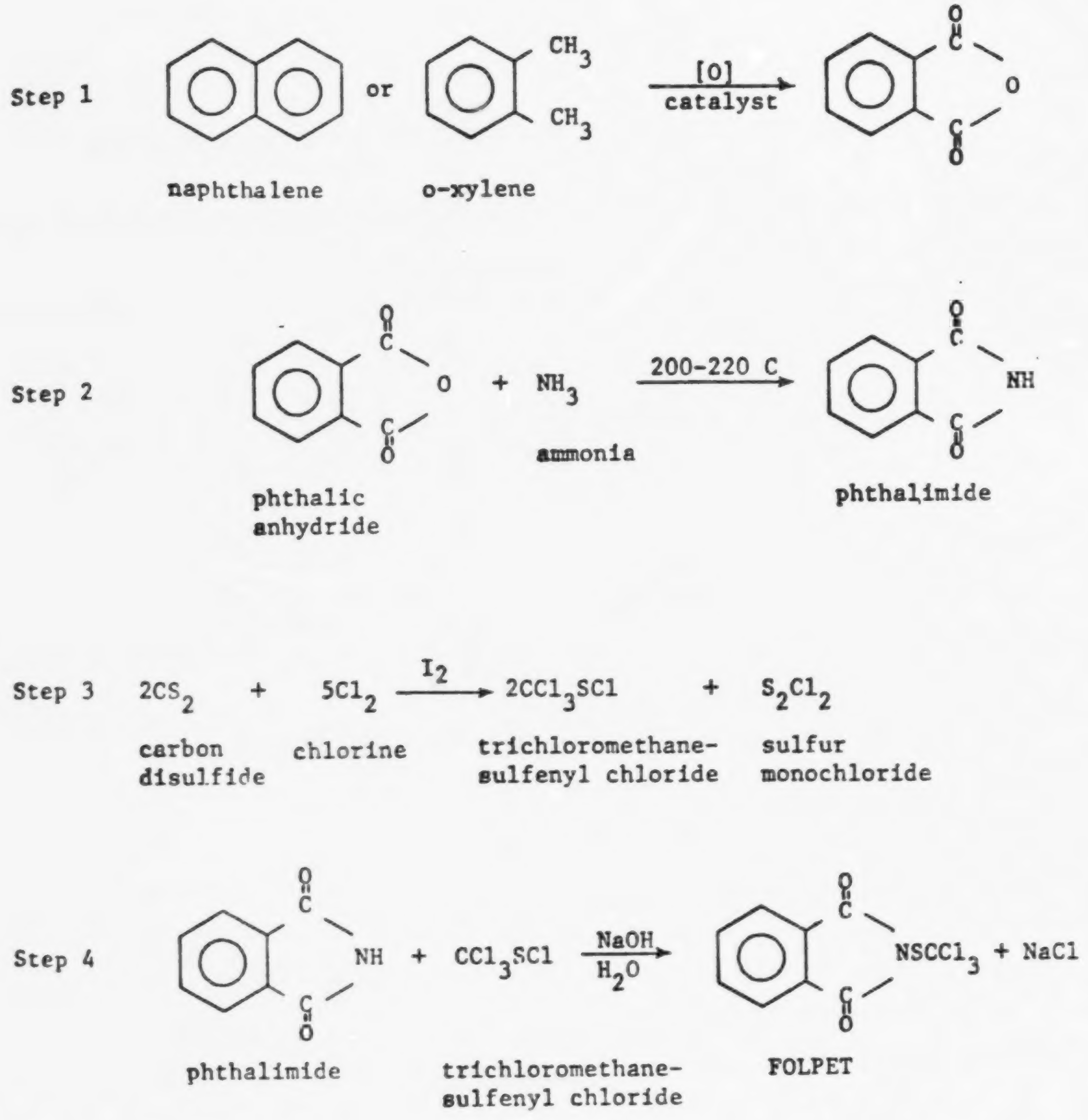

Step 1
naphthalene
or
o-xylene
CH_3
CH_3
[O]
catalyst
Step 2
phthalic anhydride
+ NH_3
ammonia
200-220 C
NH
phthalimide
Step 3
$2CS_2 + 5Cl_2 \xrightarrow{I_2} 2CCl_3SCl + S_2Cl_2$
carbon disulfide
chlorine
trichloromethane-sulfenyl chloride
sulfur monochloride
Step 4
phthalimide
+ CCl_3SCl
trichloromethane-sulfenyl chloride
NaOH
H_2O
$NSCCl_3$ + NaCl
FOLPET

FIGURE B-19. MAIN REACTIONS IN THE PREPARATION OF FOLPET

TABLE B-31. REACTIONS IN PRODUCTION AND STORAGE OF FOLPET

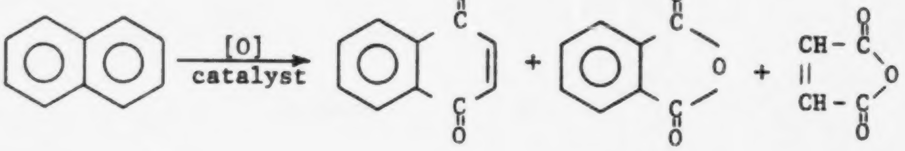
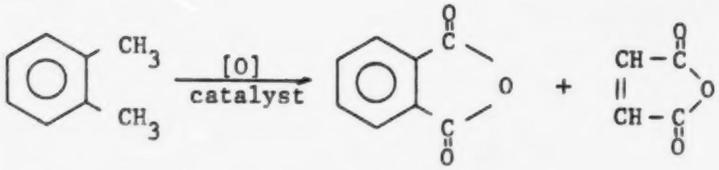
Reaction Number	Generalized Reaction
1	
2	
3	$(RCO)_2O + H_2O \longrightarrow 2\ RCOOH$
4	$(RCO)_2O + NH_3 \longrightarrow RCONH_2 + RCOOH \rightleftharpoons (RCO)_2NH + H_2O$
5	$(RCO)_2NH + NH_3 \longrightarrow 2\ RCONH_2$
6	$RCOOH + NH_3 \xrightarrow{\Delta} RCONH_2 + H_2O$
7	$2CS_2 + 3Cl_2 \xrightarrow{I_2} 2CSCl_2 + S_2Cl_2$
8	$CSCl_2 + Cl_2 \longrightarrow CCl_3SCl$
9	$CSCl_2 + 3Cl_2 + 2H_2O \longrightarrow CCl_3SO_2Cl + 4HCl$
10	$2S_2Cl_2 + 2H_2O \longrightarrow 3S + 4HCl + SO_2$

TABLE B-31 (Continued)

Reaction Number	Generalized Reaction
11	$4CCl_3SCl \xrightarrow{Fe} S_2Cl_2 + 2CCl_4 + CCl_3SSCCl_3$
12	$2CCl_3SCl + Cl_2 \longrightarrow 2CCl_4 + S_2Cl_2$
13	$CCl_3SCl + H_2O \longrightarrow CCl_3SOH + HCl$
14	$CCl_3SOH + H_2O \longrightarrow CO_2 + S + 3HCl$
15	$CCl_3SOH + CCl_3SCl \xrightarrow{NaOH} CCl_3SOSCCl_3$
16	$2CCl_3SOSCCl_3 \longrightarrow CCl_3SO_2SCCl_3 + CCl_3SSCCl_3$
17	$CCl_3SO_2SCCl_3 + H_2O \longrightarrow CCl_3SO_3H + CCl_3SH$
18	$CCl_3SO_2Cl + H_2O \longrightarrow CCl_3SO_3H + HCl$
19	$CCl_3SCl + (RCO)_2NH \xrightarrow{NaOH} (RCO)_2NSCCl_3$
20	$CCl_3SO_2Cl + (RCO)_2NH \xrightarrow{NaOH} (RCO)_2NSO_2CCl_3$
21	$CSCl_2 + (RCO)_2NH \longrightarrow (RCO)_2NCSN(COR)_2$
22	$(RCO)_2NH + S_2Cl_2 \longrightarrow (RCO)_2NSSN(COR)_2$
23	$CCl_3SCl + RCH{=}CHR \longrightarrow R(CCl_3S)CHCHClR$
24	$RCH{=}CHR + CCl_3SO_2Cl \longrightarrow RCHClCHRCCl_3 + SO_2$
25	$(RCO)_2NSCCl_3 + H_2O \longrightarrow (RCO)_2NH + CCl_3SOH$
26	$(RCO)_2NSCCl_3 \xrightarrow{[O]} (RCO)_2NSO_2CCl_3$
27	$(RCO)_2NH + 2CCl_3SCl \qquad (RCO)_2NCl + CCl_3SSCCl_3 + HCl$
28	$RCONH_2 + 2CCl_3SCl \qquad RCONHCl + CCl_3SSCCl_3 + HCl$

TABLE B-32. POSSIBLE CONTAMINANTS IN FOLPET

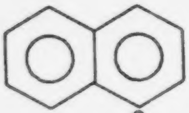
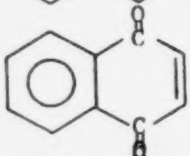
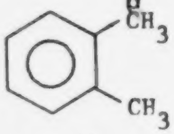
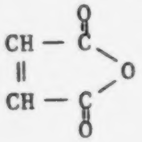
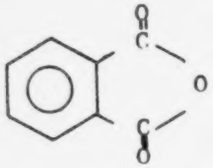
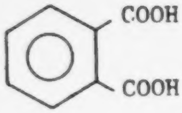
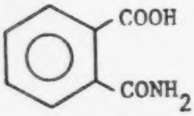
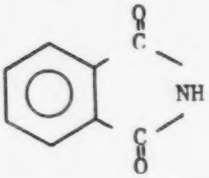
No.	Contaminant	Precursor	Reaction Number	Step	Likelihood of Being Present in Final Product
1.		SM	--	1	0
2.		1	1	1	0
3.		SM	--	1	0
4.		1 3	1 2	1 1	+
5.		1 3	1 2	1 1	++
6.	HOOCCH=CHCOOH	4	3	1 or 4	0
7.		5	3	1 or 4	+
8.		5 7	4 6	2 2	+
9.		8 26	4 25	2 St	++

TABLE B-32. (Continued)

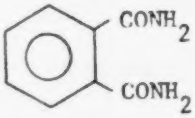
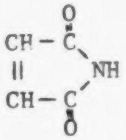
No.	Contaminant	Precursor	Reaction Number	Step	Likelihood of Being Present in Final Product
10.		9 8	5 6	2 2	+
11.	$HOOCCH=CHCONH_2$	4 6	4 6	2 2	0
12.		11	4	2	+
13.	$H_2NOCCH=CHCONH_2$	12 11	5 6	2 2	0
14.	CS_2	SM	--	3	0
15.	$CSCl_2$	14	7	3	0
16.	S_2Cl_2	14 17 17	7 11 12	3 3 3	0
17.	CCl_3SCl	15	8	3	+
18.	CCl_3SO_2Cl	15	9	3	0
19.	S	16 21	10 14	3 4 or St	++
20.	CCl_3SSCCl_3	17 22	11 16	3 4 or St	+
21.	CCl_3SOH	17 26	13 25	4 4 or St	0
22.	$CCl_3SOSCCl_3$	21+17	15	4	0
23.	$CCl_3SO_2SCCl_3$	22	16	4	+
24.	CCl_3SO_3H	23 18	17 18	4 4	+

TABLE B-32 (Continued)

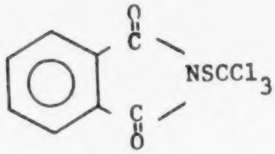
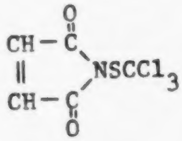
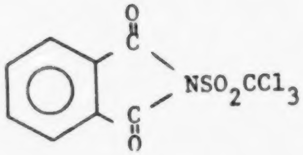
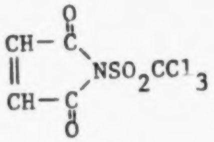
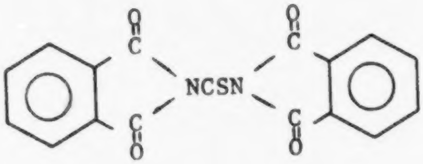
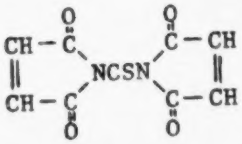
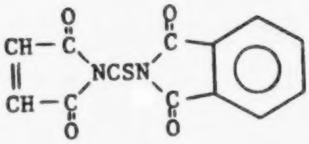
No.	Contaminant	Precursor	Reaction Number	Step	Likelihood of Being Present in Final Product
25.	CCl_3SH	23	17	4	+
26.		9+17	19	4	FOLPET
27.		12+17	19	4	++
28.		9+18 26	20 26	4 4 or St	++
29.		12+18	20	4	+
30.		9+15	21	4	+
31.		12+15	21	4	0
32.		9+12+15	21	4	0

TABLE B-32. (Continued)

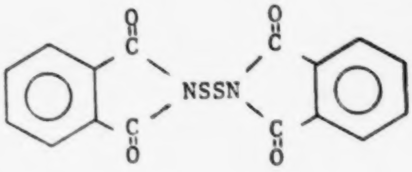
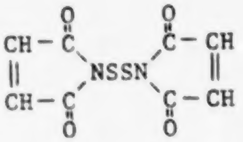
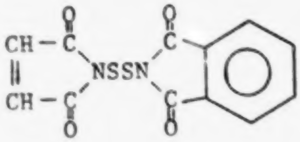
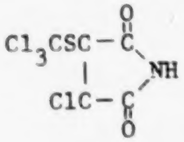
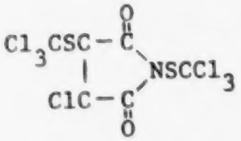
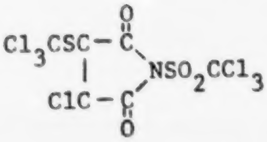
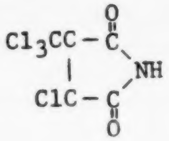
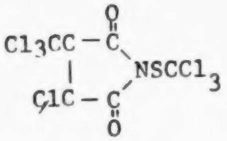
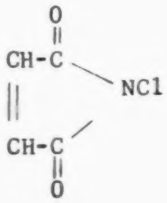
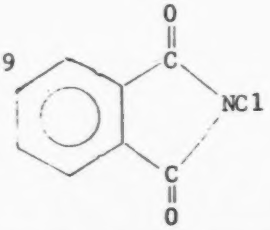
No.	Contaminant	Precursor	Reaction Number	Step	Likelihood of Being Present in Final Product
33.		9+16	22	4	+
34.		12+16	22	4	0
35.		9+12+15	22	4	0
36.		12+17	23	4	0
37.		27+17 36+17	23 19	4 4	0
38.		29+17	23	4	0
39.	$HOOCCH(SCCl_3)CHClCOOH$	6+17	23	4	0
40.	$HOOCCH(SCCl_3)CHClCONH_2$	11+17	23	4	0
41.	$H_2NOCCH(SCCl_3)CHClCONH_2$	13+17	23	4	0

TABLE B-32. (Continued)

No.	Contaminant	Precursor	Reaction Number	Step	Likelihood of Being Present in Final Product
42.		12+18	24	4	0
43.		27+18 42+17	24 19	4 4	0
44	HOOCCOOH	1 3	1 2	1 1	0
45	$HOOCCONH_2$	44	4	2	+
46	$H_2NOCCONH_2$	45	4	2	0
47	HOOCCONHCl	45	28	4	+
48		12	27	4	+
49		9	27	4	+

a. Precursor refers to the contaminant numbered in this table. SM = starting material.

b. See Table B-31.

c. The step (Figure B-19) during which the contaminant might be formed. St indicares possible formation during storage.

d. 0 = likely present only at levels below 1 ppm.
 + = likely present at levels above 1 ppm but below 100 ppm.
 ++ = likely present at levels above 100 ppm.

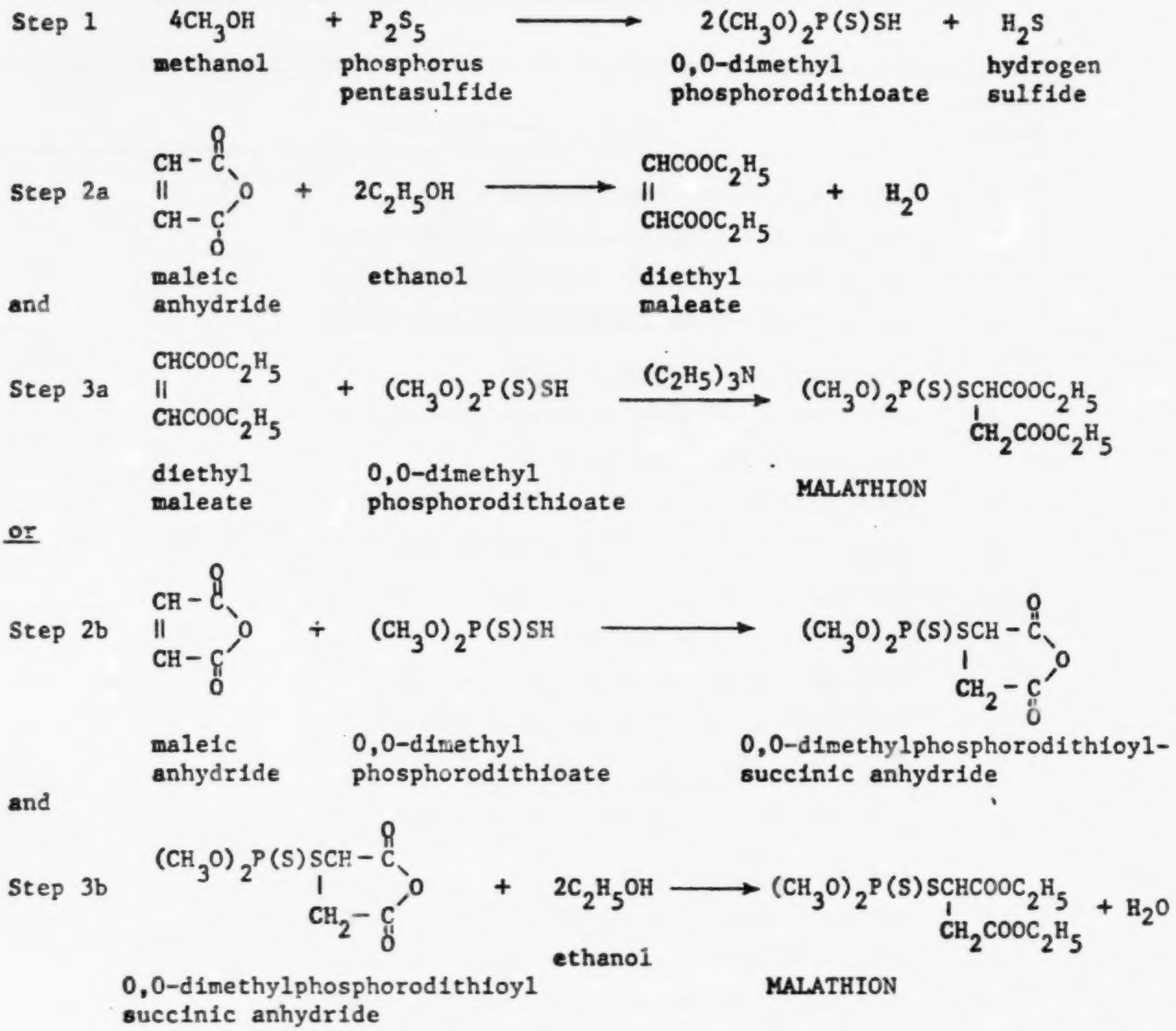

Step 1
4CH3OH
methanol
+ P2S5
phosphorus pentasulfide
2(CH3O)2P(S)SH
0,0-dimethyl phosphorodithioate
+ H2S
hydrogen sulfide
Step 2a
maleic anhydride
+ 2C2H5OH
ethanol
diethyl maleate
+ H2O
and
Step 3a
diethyl maleate
+ (CH3O)2P(S)SH
0,0-dimethyl phosphorodithioate
(C2H5)3N
(CH3O)2P(S)SCHCOOC2H5
CH2COOC2H5
MALATHION
or
Step 2b
maleic anhydride
+ (CH3O)2P(S)SH
0,0-dimethyl phosphorodithioate
0,0-dimethylphosphorodithioyl-succinic anhydride
and
Step 3b
0,0-dimethylphosphorodithioyl succinic anhydride
+ 2C2H5OH
ethanol
(CH3O)2P(S)SCHCOOC2H5
CH2COOC2H5
+ H2O
MALATHION

FIGURE B-20. SYNTHESIS OF MALATHION

TABLE B-33. R[illegible]ONS OCCURRING DURING PRODUCTION OF MALATHION

Reaction Number	Generalized Reaction[a]
1	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
7	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
8	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
9	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
10	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
11	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
12	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
13	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
14	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(YR)_2$
15	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$
16	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
17	$(RCO)_2O + R'OH \longrightarrow RCOOH + RCOOR'$
18	$RCOOH + R'OH \rightleftharpoons RCOOR' + H_2O$
19	$(RY)_2P(Y)SH + -CH=CH- \rightleftharpoons (RY)_2P(Y)S\underset{\vert}{C}HCH_2-$
20	$(RCO)_2O + H_2O \longrightarrow 2RCOOH$
21	$RSH + -CH=CH- \longrightarrow RS\underset{\vert}{C}HCH_2-$
22	$H_2S + -CH=CH= \longrightarrow HS\underset{\vert}{C}HCH_2-$
23	$2RSH \xrightarrow{[O]} RSSR$
24	$RCOOR' + R''OH \longrightarrow RCOOR'' + R'OH$
25	$(RY)_2P(Y)OR + R'OH \longrightarrow (RY)_2P(Y)(OR') + ROH$

a. R = alkyl or substituted alkyl group; Y = oxygen or sulfur.

TABLE B-34. POSSIBLE CONTAMINANTS IN MALATHION

No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	P_2S_5	SM	--	1	0
2	CH_3OH	SM	--	1	+
3	$(CH_3O)_2P(S)SH$	1+2	1	1	++
4	$(CH_3O)_2(CH_3S)PS$	3+2	2	1	++
5	$(CH_3O)_3PS$	3+2 4	3 4	1 1	++
6	$(CH_3O)(CH_3S)_2PS$	4 4	5 4	1 1	+
7	$(CH_3O)(CH_3S)P(S)SH$	3 3	5 4	1 1	+
8	$(CH_3O)_2P(S)OH$	3 5 30	6 15 15	1 1 St	+
9	$(CH_3O)_2P(O)SH$	8	7	1 or St	+
10	$(CH_3O)_2P(O)OH$	9	6	1 or St	+
11	$(CH_3O)(CH_3S)P(O)SH$	7	7	1	0
12	$(CH_3S)_2P(S)SH$	7 7	5 4	1 1	0
13	$(CH_3O)_2P(S)SP(S)(OCH_3)_2$	3	8	1	+
14	$(CH_3O)_2P(S)SSP(S)(OCH_3)_2$	3	9	1	0
15	$(CH_3O)_2PSH$	3	11	1	0
16	$(CH_3O)_3P$	15	12	1	0
17	$(CH_3O)_2(CH_3S)P$	15	13	1	0
18	$(CH_3O)_2P(S)SP(OCH_3)_2$	15	14	1	0
19	$(CH_3O)_2(CH_3S)PO$	5	10	1	+

TABLE B-34. (Continued)

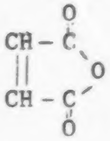
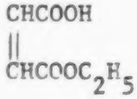
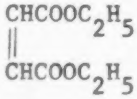
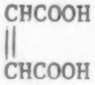
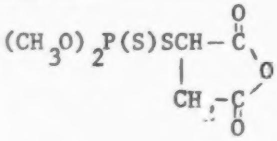
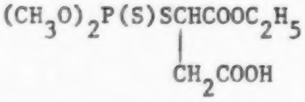
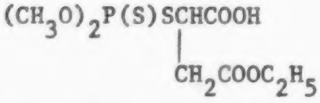
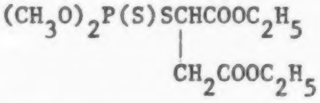
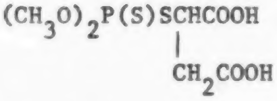
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
20	$(CH_3O)_3PO$	19 5	4 16	1	0
21	$(CH_3S)_3PS$	6	4	1	0
22		SM	--	2a,2b	++
23	C_2H_5OH	SM	--	2a,3b	+
24	 $\begin{array}{l}\text{CHCOOH}\\ \|\\ \text{CHCOOC}_2\text{H}_5\end{array}$	22+23	17	2a	+
25	 $\begin{array}{l}\text{CHCOOC}_2\text{H}_5\\ \|\\ \text{CHCOOC}_2\text{H}_5\end{array}$	24+23	18	2a	++
26	 $\begin{array}{l}\text{CHCOOH}\\ \|\\ \text{CHCOOH}\end{array}$	24 22	18 18	2a 2a	+
27		3+22	19	2b	++
28	 $\begin{array}{l}(\text{CH}_3\text{O})_2\text{P(S)SCHCOOC}_2\text{H}_5\\ \qquad\qquad\quad\ \ \|\\ \qquad\qquad\quad\ \text{CH}_2\text{COOH}\end{array}$	3+24 27+23	19 17	3a 3b	++
29	 $\begin{array}{l}(\text{CH}_3\text{O})_2\text{P(S)SCHCOOH}\\ \qquad\qquad\quad\ \ \|\\ \qquad\qquad\quad\ \text{CH}_2\text{COOC}_2\text{H}_5\end{array}$	3+24 27+23	19 17	3a 3b	++
30	 $\begin{array}{l}(\text{CH}_3\text{O})_2\text{P(S)SCHCOOC}_2\text{H}_5\\ \qquad\qquad\quad\ \ \|\\ \qquad\qquad\quad\ \text{CH}_2\text{COOC}_2\text{H}_5\end{array}$	28+23 29+23 3+25	18 18 19	3b 3b 3a	MALATHION
31	 $\begin{array}{l}(\text{CH}_3\text{O})_2\text{P(S)SCHCOOH}\\ \qquad\qquad\quad\ \ \|\\ \qquad\qquad\quad\ \text{CH}_2\text{COOH}\end{array}$	3+26 28 29	19 18 18	2b	+
32	CH_3SH	4	4	1	+

TABLE B-34. (Continued)

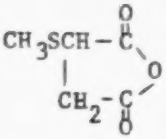
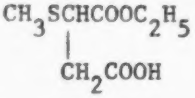
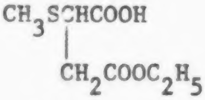
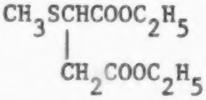
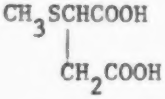
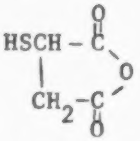
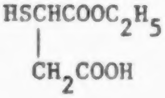
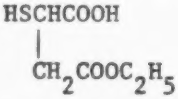
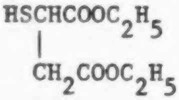
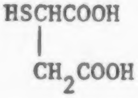
No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
33		32+22	21	2b	0
34	 $\begin{array}{l} CH_3SCHCOOC_2H_5 \\ \quad\; | \\ \quad\; CH_2COOH \end{array}$	32+24 33+23	21 17	3a 3b	+
35	 $\begin{array}{l} CH_3SCHCOOH \\ \quad\; | \\ \quad\; CH_2COOC_2H_5 \end{array}$	32+24 33+23	21 17	3a 3b	+
36	 $\begin{array}{l} CH_3SCHCOOC_2H_5 \\ \quad\; | \\ \quad\; CH_2COOC_2H_5 \end{array}$	32+25 34+23 35+23	21 17 17	3a 3b 3b	+
37	 $\begin{array}{l} CH_3SCHCOOH \\ \quad\; | \\ \quad\; CH_2COOH \end{array}$	32+26 33	21 20	2b 3b	0
38	H_2S	1+2	1	1	0
39		38+22	22	2b	0
40	 $\begin{array}{l} HSCHCOOC_2H_5 \\ | \\ CH_2COOH \end{array}$	38+24 39+23	22 17	3a 3b	+
41	 $\begin{array}{l} HSCHCOOH \\ | \\ CH_2COOC_2H_5 \end{array}$	38+24 39+23	22 17	3a 3b	+
42	 $\begin{array}{l} HSCHCOOC_2H_5 \\ | \\ CH_2COOC_2H_5 \end{array}$	38+25 40+23 41+23	22 17 17	3a 3b 3b	+
43	 $\begin{array}{l} HSCHCOOH \\ | \\ CH_2COOH \end{array}$	38+26 39	22 20	2b 3b	0

TABLE B-34. (Continued)

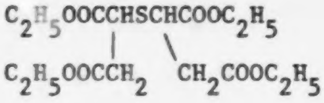
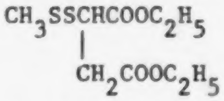
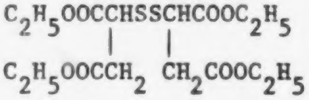
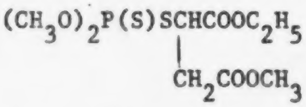
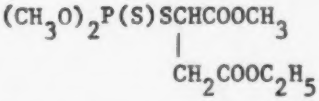
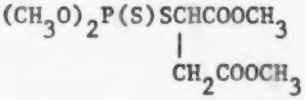
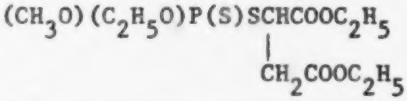
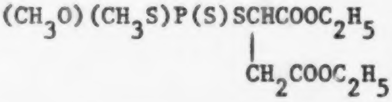
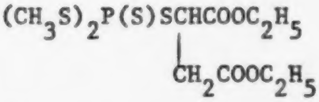
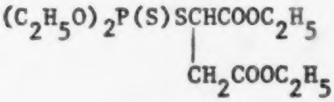
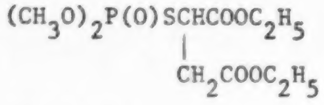
No.	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
44	 $\begin{array}{l} C_2H_5OOCCHSCHCOOC_2H_5 \\ \qquad\quad | \quad \backslash \\ C_2H_5OOCCH_2 \ \ CH_2COOC_2H_5 \end{array}$	42+25	21	3a	+
45	CH_3SSCH_3	32	23	3a,2b	+
46	 $\begin{array}{l} CH_3SSCHCOOC_2H_5 \\ \qquad | \\ \qquad CH_2COOC_2H_5 \end{array}$	32+42	23	3a,2b	0
47	 $\begin{array}{l} C_2H_5OOCCHSSCHCOOC_2H_5 \\ \qquad\quad | \quad\ \ | \\ C_2H_5OOCCH_2 \ \ CH_2COOC_2H_5 \end{array}$	42	23	3a,3b	0
48	 $\begin{array}{l} (CH_3O)_2P(S)SCHCOOC_2H_5 \\ \qquad\qquad\quad | \\ \qquad\qquad\quad CH_2COOCH_3 \end{array}$	30+2	24	3a,3b	+
49	 $\begin{array}{l} (CH_3O)_2P(S)SCHCOOCH_3 \\ \qquad\qquad\quad | \\ \qquad\qquad\quad CH_2COOC_2H_5 \end{array}$	30+2	24	3a,3b	+
50	 $\begin{array}{l} (CH_3O)_2P(S)SCHCOOCH_3 \\ \qquad\qquad\quad | \\ \qquad\qquad\quad CH_2COOCH_3 \end{array}$	48+2 49+2	24 24	3a,3b 3a,3b	0
51	 $\begin{array}{l} (CH_3O)(C_2H_5O)P(S)SCHCOOC_2H_5 \\ \qquad\qquad\qquad\qquad | \\ \qquad\qquad\qquad\qquad CH_2COOC_2H_5 \end{array}$	30+23	25	3a,3b	+
52	 $\begin{array}{l} (CH_3O)(CH_3S)P(S)SCHCOOC_2H_5 \\ \qquad\qquad\qquad\quad | \\ \qquad\qquad\qquad\quad CH_2COOC_2H_5 \end{array}$	7+25	19	3a	++
53	 $\begin{array}{l} (CH_3S)_2P(S)SCHCOOC_2H_5 \\ \qquad\qquad\quad | \\ \qquad\qquad\quad CH_2COOC_2H_5 \end{array}$	12+25	19	3a	+
54	 $\begin{array}{l} (C_2H_5O)_2P(S)SCHCOOC_2H_5 \\ \qquad\qquad\quad | \\ \qquad\qquad\quad CH_2COOC_2H_5 \end{array}$	51+23	25	3a,3b	0

TABLE B-34. (Continued)

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
55	 $(CH_3O)_2P(O)S\underset{CH_2COOC_2H_5}{CH}COOC_2H_5$	30	16	St	+
56	$(C_2H_5)_3N$	Cat	19	3a	+

a. Precursor refers to the contaminant numbered in this table. SM = starting material.

b. See Table B-33.

c. The step (Figure B-20) during which the contaminant might be formed. St indicates possible formation during storage.

d. 0 = likely present only at levels below 1 ppm.
+ = likely present at levels above 1 ppm but below 100 ppm.
++ = likely present at levels above 100 ppm.

Step 1

$$CH_3CHO + NH_2OH \longrightarrow CH_3CH{=}NOH$$

acetaldehyde	hydroxylamine	acetaldehyde oxime

Step 2

$$CH_3CH{=}NOH + Cl_2 \xrightarrow{DMF} CH_3C(Cl){=}NOH + HCl$$

acetaldehyde oxime	chlorine	dimethylformamide	N-hydroxyethanimidoyl chloride	hydrogen chloride

Step 3

$$CH_3C(Cl){=}NOH + CH_3SH \xrightarrow{NaOH} CH_3C(SCH_3){=}NOH$$

N-hydroxyethanimidoyl chloride	methyl mercaptan	sodium hydroxide	methyl N-hydroxyethanimidothioate

Step 4

$$CH_3C(SCH_3) + NOH + CH_3NCO \xrightarrow{(C_2H_5)_3N} CH_3C(SCH_3){=}NOCONHCH_3$$

methyl-N-hydroxyethanimidothioate	methyl isocyanate	triethyl amine	methyl N-[[(methylamino)-carbonyl]oxy] ethanimidothioate (methomyl)

FIGURE B-21. SYNTHESIS OF METHOMYL

TABLE B-35. REACTIONS OCCURRING DURING PRODUCTION AND STORAGE OF METHOMYL

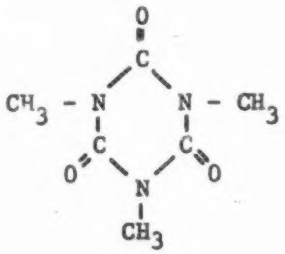
Reaction Number	Reaction
1	$CH_3CHO + NH_2OH \longrightarrow CH_3CH{=}NOH + H_2O$
2	$CH_3CH{=}NOH + Cl_2 \xrightarrow{DMF} CH_3C(Cl){=}NOH + HCl$
3	$CH_3C(Cl){=}NOH \rightleftarrows CH_3CH(Cl)NO \xrightarrow{Cl_2} CH_3CCl_2NO + HCl$
4	$CH_3CH{=}NOH + Cl_2 \longrightarrow CH_2ClCH{=}NOH + HCl$
5	$CH_3C(Cl){=}NOH + Cl_2 \longrightarrow CH_2ClC(Cl){=}NOH + HCl$
6	$CH_3C(Cl){=}NOH + H_2O \longrightarrow [CH_3C(OH){=}NOH] \longrightarrow CH_3CONHOH + HCl$
7	$CH_2CLC(Cl){=}NOH + H_2O \longrightarrow CH_2ClCONHOH$
8	$CH_3C(Cl){=}NOH + CH_3SH \xrightarrow{NaOH} CH_3C(SCH_3){=}NOH + NaCl$
9	$2CH_3SH \xrightarrow{[O]} CH_3SSCH_3$
10	$CH_2ClC(Cl){=}NOH + CH_3SH \longrightarrow CH_2ClC(SCH_3){=}NOH$
11	$CH_2ClC(Cl){=}NOH + 2CH_3SH \xrightarrow{NaOH} CH_3SCH_2C(SCH_3){=}NOH$
12	$CH_2ClCONHOH + CH_3SH \xrightarrow{NaOH} CH_3SCH_2CONHOH$
13	$CH_3NCO + H_2O \longrightarrow [CH_3NHCOOH] \longrightarrow CH_3NH_2 + CO_2$
14	$3CH_3NCO \longrightarrow$ 
15	$CH_3NCO + CH_3NH_2 \longrightarrow CH_3NHCONHCH_3$
16	$CH_3C(SCH_3){=}NOH + CH_3NCO \longrightarrow CH_3C(SCH_3){=}NOCONHCH_3$
17	$CH_3C(Cl){=}NOH + CH_3NCO \longrightarrow CH_3C(Cl){=}NOCONHCH_3$

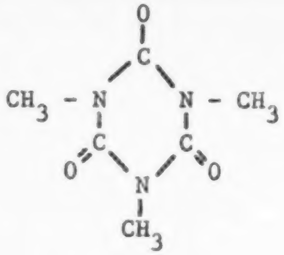
TABLE B-35. (Continued)

Reaction Number	Reaction
18	$CH_3NCO + CH_3SH \longrightarrow CH_3NHCOSCH_3$
19	$CH_3CH{=}NOH + CH_3NCO \longrightarrow CH_3CH{=}NOCONHCH_3$
20	$CH_3C(SCH_3){=}NOCONHCH_3 \xrightarrow{[O]} CH_3C(SOCH_3){=}NOCONHCH_3$
21	$CH_3C(SOCH_3){=}NOCONHCH_3 \xrightarrow{[O]} CH_3C(SO_2CH_3){=}NOCONHCH_3$
22	$CH_3C(SCH_3){=}NOCONHCH_3 + H_2O \longrightarrow CH_3C(SCH_3){=}NOH + CO_2 + CH_3NH_2$
23	$CH_3C(SCH_3){=}NOH + H_2O \longrightarrow CH_3SH + [CH_3C(OH){=}NOH] \longrightarrow CH_3CONHOH$
24	$CH_3CONHOH + H_2O \longrightarrow CH_3COOH + NH_2OH$

TABLE B-36. POSSIBLE CONTAMINANTS IN METHOMYL

Number	Contaminant	Precursor(a)	Reaction Number(b)	Step(c)	Likelihood of Being Present in Final Product(d)
1	CH_3CHO	SM 10	-- 24	1 St	0
2	NH_2OH	SM	--	1	0
3	$CH_3CH{=}NOH$	1 + 2	1	1	0
4	Cl_2	SM	--	2	0
5	$(CH_3)_2NCHO$ (DMF)	Solv.	2	2	0
6	$CH_3C(Cl){=}NOH$	3 + 4	2	2	0
7	CH_3CCl_2NO	4 + 6	3	2	0
8	$CH_2ClCH{=}NOH$	3 + 4	4	2	0
9	$CH_2ClC(Cl){=}NOH$	4 + 6	5	2	0
10	$CH_3CONHOH$	6 23	6 22, 23	3 St	++
11	$CH_2ClCONHOH$	9	7	3	0
12	CH_3SH	SM 23	-- 22, 23	3 St	+
13	$CH_3C(SCH_3){=}NOH$	6 + 12 23	8 22	3 St	++
14	CH_3SSCH_3	12	9	3	+
15	$CH_2ClC(SCH_3){=}NOH$	9 + 12	10	3	0
16	$CH_3SCH_2C(SCH_3){=}NOH$	9 + 12	11	3	0
17	$CH_3SCH_2CONHOH$	11 + 12	12	3	0
18	CH_3NCO	SM	--	4	0
19	$(C_2H_5)_3N$	Cat.	--	4	0
20	CH_3NH_2	18 23	13 22	4 St	+

TABLE B-36. (Continued)

Number	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product[(d)]
21		18	14	4	++
22	$CH_3NHCONHCH_3$	18 + 20	15	4	+
23	$CH_3C(SCH_3)=NOCONHCH_3$	13 + 18	16	4	Methomyl
24	$CH_3C(Cl)=NOCONHCH_3$	6 + 18	17	4	++
25	$CH_3NHCOSCH_3$	12 + 18	18	4	+
26	$CH_3CH=NOCONHCH_3$	3 + 18	19	4	++
27	$CH_3C(SOCH_3)=NOCONHCH_3$	23	20	St	0
28	$CH_3C(SO_2CH_3)=NOCONHCH_3$	27	21	St	0
29	CH_3COOH	10	24	St	0

(a) Precursor refers to the contaminant numbered in this table.
SM = starting material; Solv. = solvent; Cat. = catalyst.

(b) See Table B-35.

(c) The step (Figure B-21) during which the contaminant might be formed.
St. indicates possible formation during storage.

(d) 0 = likely to be present at levels below 1 ppm

+ = likely to be present at levels above 1 ppm but below 100 ppm

++ = likely to be present at levels above 100 ppm.

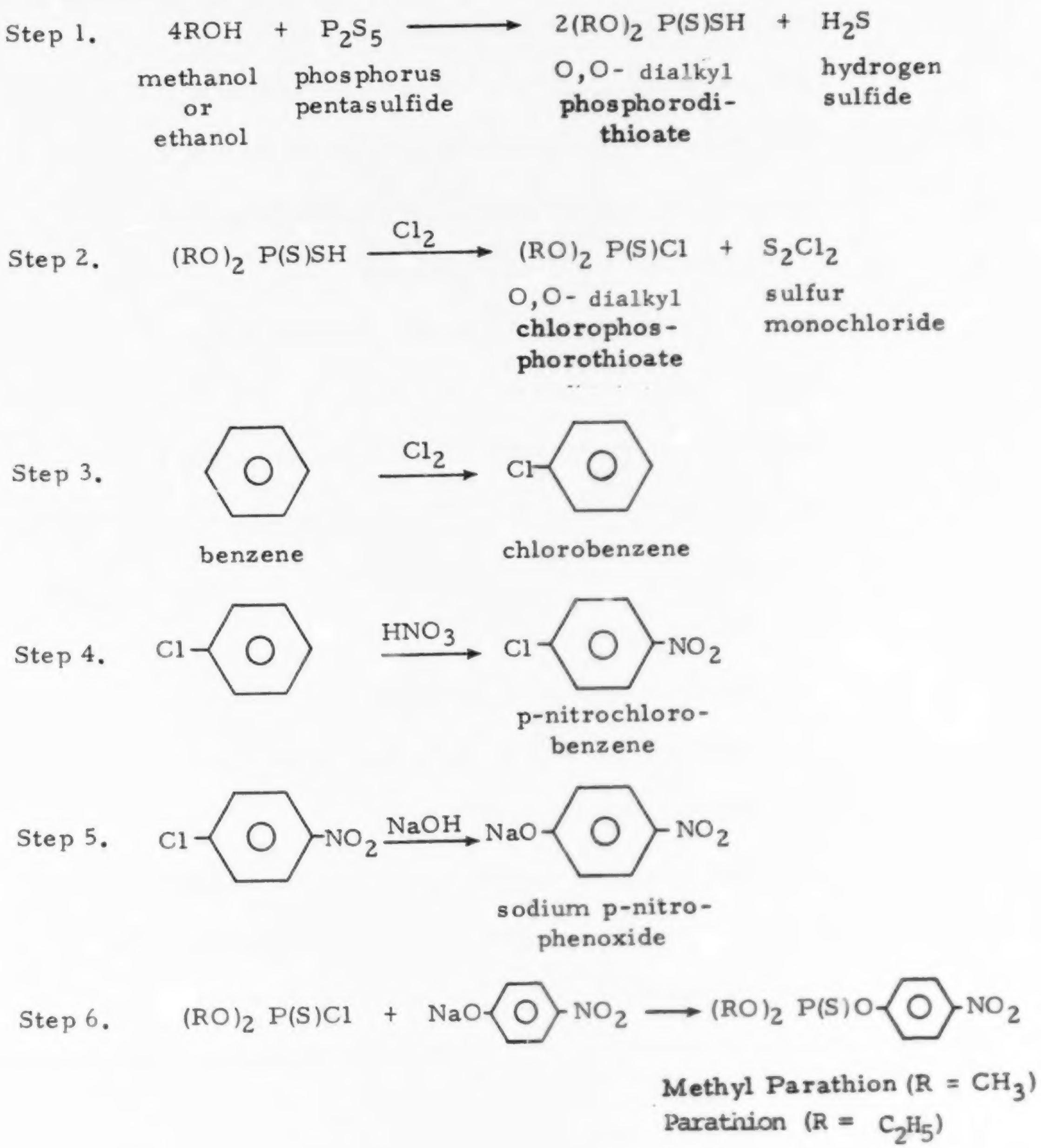

Step 1.
4ROH + P2S5 → 2(RO)2 P(S)SH + H2S
methanol or ethanol
phosphorus pentasulfide
O,O- dialkyl phosphorodithioate
hydrogen sulfide
Step 2.
(RO)2 P(S)SH
Cl2
(RO)2 P(S)Cl + S2Cl2
O,O- dialkyl chlorophosphorothioate
sulfur monochloride
Step 3.
benzene
Cl2
Cl
chlorobenzene
Step 4.
Cl
HNO3
Cl
NO2
p-nitrochlorobenzene
Step 5.
Cl
NO2
NaOH
NaO
NO2
sodium p-nitrophenoxide
Step 6.
(RO)2 P(S)Cl + NaO
NO2
(RO)2 P(S)O
NO2
Methyl Parathion (R = CH3)
Parathion (R = C2H5)

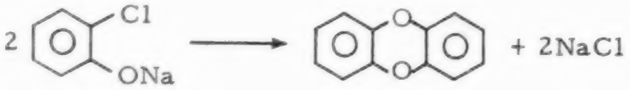
FIGURE B-22. MAIN REACTIONS IN THE PREPARATION OF METHYL PARATHION AND PARATHION

TABLE B-37. REACTIONS OCCURRING DURING PRODUCTION OF PARATHION AND METHYL PARATHION

Reaction Number	Generalized Reaction$^{(a)}$
1.	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2.	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3.	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4.	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5.	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6.	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
7.	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
8.	$(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
9.	$(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
10.	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
11.	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
12.	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
13.	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
14.	$(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(YR)_2$
15.	$(RY)_2PSH + Cl_2 \longrightarrow (RY)_2P(S)SP(YR)_2 + 2HCl$
16.	$(RY)_2P(S)SH + Cl_2 \longrightarrow (RY)_2P(S)SSP(S)(YR)_2 + 2HCl$
17.	$(RY)_2P(S)SSP(S)(YR)_2 + Cl_2 \longrightarrow 2(RY)_2P(S)Cl + S_2Cl_2$

(a) R = alkyl or aryl group; Ar = aryl group; Y = oxygen or sulfur.

TABLE B-37. (Continued)

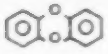
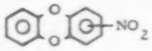
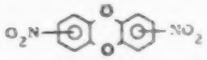
Reaction Number	Generalized Reaction[(a)]
18.	$2S_2Cl_2 + 2H_2O \longrightarrow SO_2 + 4HCl + S$
19.	$ArH \xrightarrow{Cl_2} ArCl$
20.	$ArH \xrightarrow{HNO_3} ArNO_2$
21.	$ArCl \xrightarrow{NaOH} ArONa \xrightarrow{H^+} ArOH$
22.	$ArCl + ROH \longrightarrow ArOR + HCl$
23.	$ArCl + ArONa \longrightarrow ArOAr + NaCl$
24.	
25.	$(RY)_2P(Y)Cl + ArONa \longrightarrow (RY)_2P(Y)OAr + NaCl$
26.	$(RY)_2P(Y)OAr + ArONa \longrightarrow (RY)(ArO)P(Y)OAr + RONa$
27.	$(RY)_3PY + ArONa \longrightarrow (RY)_2P(Y)YNa + ArOR$
28.	$(RY)_2P(Y)Cl + (RY)_2P(Y)YNa \longrightarrow (RY)_2P(Y)YP(Y)(YR)_2 + NaCl$
29.	$(RY)_2P(Y)Cl + ROH \longrightarrow (RY)_2(OR)PY + HCl$
30.	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
31.	$(RY)_3PY + H_2O \longrightarrow (RY)_2P(Y)OH + RYH$
32.	$RSH \xrightarrow{[O]} RSSR$

(a) R = alkyl or aryl group; Ar = aryl group; Y = oxygen or sulfur.

TABLE B-38. POSSIBLE CONTAMINANTS IN PARATHION AND METHYL PARATHION

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
1	P_2S_5	SM	--	1	o
2	$(RO)_2P(S)SH$	1	1	1	o
3	$(RO)_2(RS)PS$	2 19	2 29	1 6	++
4	$(RO)_3PS$	2 3 18	3 4 29	1 1 6	++
5	$(RO)(RS)_2PS$	3	5	1	++
6	$(RO)(RS)P(S)SH$	2	5	1	o
7	$(RO)_2P(O)OH$	7	6	1	o
8	$(RO)_2P(O)SH$	2	6 + 7	1	o
9	$(RO)(RS)P(O)SH$	6	6 + 7	1	o
10	$(RO)_2P(S)SP(S)(OR)_2$	2	9	1	++
11	$(RO)_2P(S)SSP(S)(OR)_2$	2 2	10 16	1 2	++
12	$(RO)_2PSH$	2	11	1	o
13	$(RO)_3P$	12	12	1	+
14	$(RO)_2(RS)P$	12	13	1	+
15	$(RO)_2P(S)SP(OR)_2$	12 12	14 15	1 2	o
16	$(RO)_2(RS)PO$	4 9 21	10 3 29	1 1 6	++
17	$(RO)_3PO$	16 20	4 29	1 6	++
18	$(RO)_2P(S)Cl$	11	17	2	o
19	$(RO)(RS)P(S)Cl$	6	16 + 17	2	o
20	$(RO)_2P(O)Cl$	8	16 + 17	2	o
21	$(RO)(RS)P(O)Cl$	9	16 + 17	2	o
22	S_2Cl_2	11	17	2	o
23	S	22	18	2	++
24	C_6H_6	SM	--	3	o
25	C_6H_5Cl	24	19	3	o
26	$C_6H_4Cl_2$	25	19	3	o
27	$C_6H_4(NO_2)Cl$	24	20	4	+
28	$C_6H_3(NO_2)_2Cl$	27	20	4	o
29	$C_6H_3(NO_2)Cl_2$	26	20	4	o
30	$C_6H_2(NO_2)_2Cl_2$	29	20	4	o
31	$C_6H_4(OH)Cl$	26	21	5	o
32	$C_6H_4(NO_2)OH$	27 48	21 31	5 St	++
33	$C_6H_3(NO_2)_2OH$	28	21	5	+

TABLE B-38. (Continued)

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
34	$C_6H_3(NO_2)(OH)Cl$	29	21	5	o
35	$C_6H_2(NO_2)_2(OH)Cl$	30	21	5	o
36	$C_6H_4(NO_2)OC_6H_4NO_2$	27 + 32	23	5	+
37	$C_6H_3(NO_2)_2OC_6H_4NO_2$	28 + 32 27 + 33	23 23	5 5	o
38	$C_6H_3(NO_2)ClOC_6H_4NO_2$	29 + 32 27 + 34	23 23	5 5	o
39	$C_6H_4ClOC_6H_4NO_2$	26 + 32 27 + 31	23 23	5 5	o
40		31	24	5	o
41		31 + 34	24	5	o
42		34	24	5	o
43	$C_6H_4(OR)Cl$	26	22 or 27	6	o
44	$C_6H_4(NO_2)OR$	27	22 or 27	6	++
45	$C_6H_3(NO_2)_2OR$	28	22 or 27	6	+
46	$C_6H_3(NO_2)(OR)Cl$	29	22 or 27	6	o
47	$C_6H_2(NO_2)_2(OR)Cl$	30	22 or 27	6	o
48	$(RO)_2P(S)OC_6H_4NO_2$	18 + 32	25	6	Parathion or Methyl Parathion
49	$(RO)_2P(S)OC_6H_3(NO_2)_2$	18 + 33	25	6	+
50	$(RO)_2P(S)OC_6H_3NO_2Cl$	18 + 34	25	6	o
51	$(RO)P(S)(OC_6H_4NO_2)_2$	48	26	6	+
52	$(RO)(RS)P(S)OC_6H_4NO_2$	19 + 32	25	6	++
53	$(RO)_2P(O)OC_6H_4NO_2$	20 + 32 48	25 30	6 St	++
54	$(RO)(RS)P(O)OC_6H_4NO_2$	21 + 32 48	25 10	6 6 or St	++
55	$(RO)_2P(O)SC_6H_4NO_2$	48	10	6 or St	+
56	$(RO)(NO_2C_6H_4O)P(S)OH$	48	31	St	o
57	$(RO)(NO_2C_6H_4O)P(O)OH$	56	7 + 6	St	+
58	$(RO)_2P(S)OH$	48	31	St	+
59	$(RO)_2P(O)OH$	58	7 + 6	St	++
60	RSH	3 or 52	31	St	+
62	RSSR	60	32	St	+

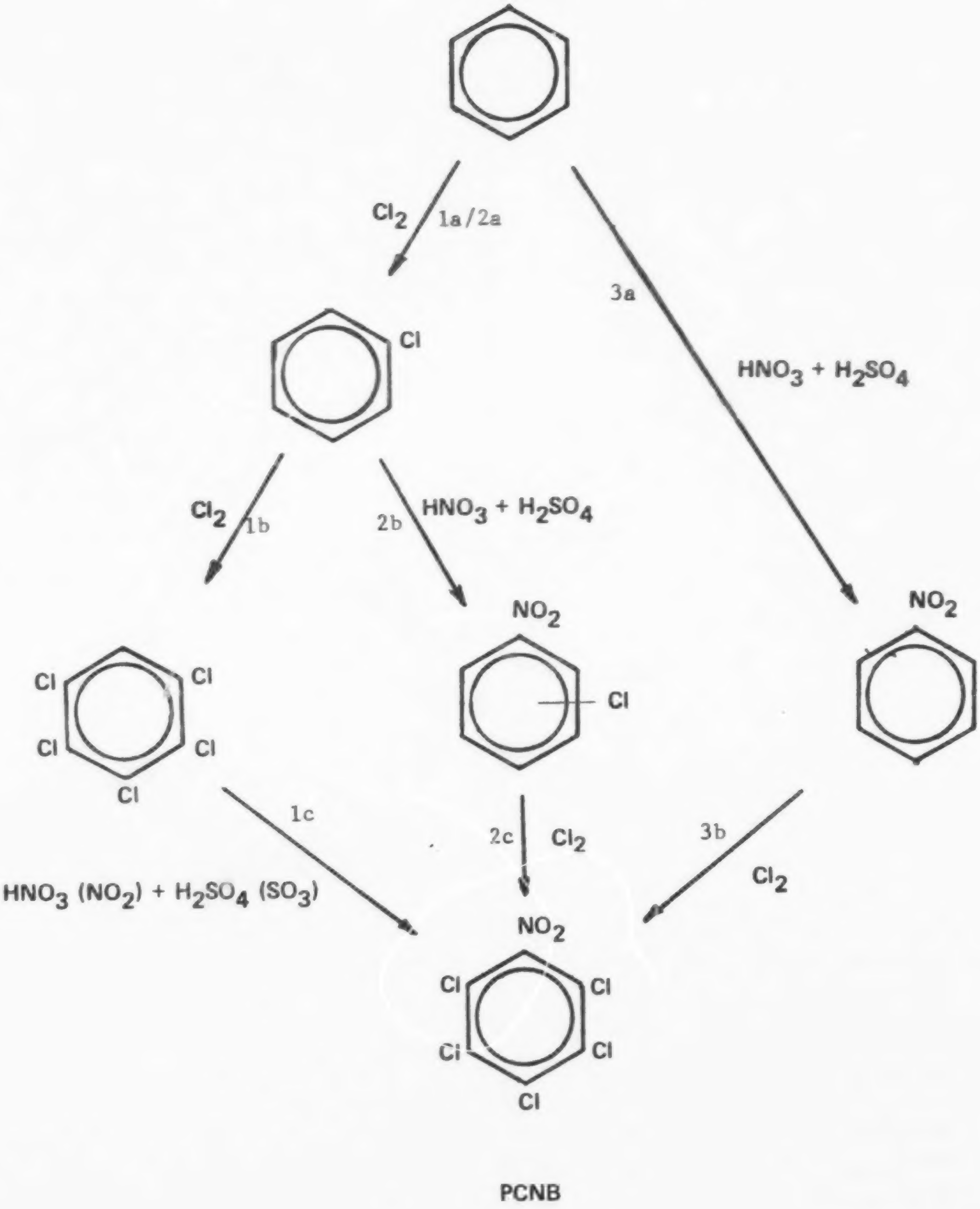
(a) R = CH_3 or C_2H_5

(b) Precursor refers to the contaminant numbered in this table. SM = starting material.

(c) See Table B-37

(d) The step (Figure B-22) during which the contaminant might be formed. St indicates possible formation during storage.

(e) o = likely present only at levels below 1 ppm
+ = likely present at levels above 1 ppm but below 100 ppm
++ = likely present at levels above 100 ppm.



Cl2
1a/2a
3a
Cl
HNO3 + H2SO4
Cl2
1b
2b
HNO3 + H2SO4
NO2
NO2
Cl
Cl
Cl
Cl
Cl
Cl
1c
2c
Cl2
3b
Cl2
HNO3 (NO2) + H2SO4 (SO3)
NO2
Cl
Cl
Cl
Cl
Cl
PCNB

FIGURE B-23. SYNTHESIS OF PCNB

TABLE B-39. POSSIBLE CONTAMINANTS IN PCNB

No.	Contaminant	Precursor$^{(a)}$	Formation Step$^{(b)}$	Likelihood of being Present in Final Product (c)
1	Benzene	SM^{d}	1a/2a,3a	0
2	Chlorobenzene	1	1a/2a,3b	0
3	Dichlorobenzenes (3 isomers)	2	1b,1a/2a,2c,3b	0
4	Trichlorobenzenes (3 isomers)	3	1b,1a/2a,2c,3b	0
5	Tetrachlorobenzenes (3 isomers)	4	1b,2c,3b	+
6	Pentachlorobenzene	5	1b,2c,3b	++
7	Hexachlorobenzene	6	1b,2c,3b	++
8	Nitrobenzene	1	2b,3a	0
9	m-Dinitrobenzene	8	2b,3a	0
10	Chloronitrobenzenes (3 isomers)	2	2b	0
		8	3b	
11	Dichloronitrobenzenes (6 isomers)	3	2b	0
		10	2c,3b	
12	Trichloronitrobenzenes (6 isomers)	4	1c,2b,	+
		11	2c,3b	
13	Tetrachloronitrobenzenes (3 isomers)	5	1c	++
		12	2c,3b	
14	Chloro-m-dinitrobenzenes (3 isomers)	9	2c,3b	0
		10	2b	
15	Dichloro-m-dinitrobenzenes (4 isomers)	11	2b	+
		14	2c,3b	
16	Trichloro-m-dinitrobenzene	12	1c,2b	+
			2c,3b	
17	Tetrachloro-m-dinitrobenzene	13	1c	++
		16	2c,3b	
18	Pentachlorobenzenesulfonic acid	6	1c	+
19	Tetrachloronitrobenzenesulfonic acid	e	e	+
20	Octachlorodibenzodioxin	5	1b	0

TABLE B-39. (Continued)

No.	Contaminant	Precursor[a]	Formation Step[b]	Likelihood of being Present in Final Product[c]
21	Octachlorodibenzofuran	5	1b	+
22	Decachlorobiphenyl	6	1b	+
23	Octachlorobiphenylene	5	1b	0
24	Hexachlorodinitrodibenzodioxin	16	2c,3b	0
25	Hexachlorodinitrodibenzofuran	16	2c,3b	+
26	Octachlorodinitrobiphenyl	17	2c,3b	+
27	Hexachlorodinitrobiphenylene	16	2c,3b	0
28	Decachlorodiphenyl ether	6	1b	0
29	Octachlorodinitrodiphenyl ether	17	2c,3b	0

a. Contaminant listed in this table.

b. Refers to the various steps of the three methods given in Figure B-23.

c. 0 = Likely present only at levels below 1 ppm

+ = Likely present at levels above 1 ppm but below 100 ppm

++ = Likely present at levels above 100 ppm.

d. Starting material.

e. Formed by various sequences of reactions in methods 2 and 3.

Step 1

$$4C_2H_5OH + P_2S_5 \longrightarrow 2(C_2H_5O_2)P(S)SH + H_2S$$

ethanol	phosphorus pentasulfide	O, O-diethyl phosphorodithioate	hydrogen sulfide

Step 2

$$(C_2H_5O)_2P(S)SH + HCHO \longrightarrow (C_2H_5O)_2P(S)SCH_2OH$$

O, O-diethyl phosphorodithioate	formaldehyde	O, O-diethyl-S-(hydroxymethyl) phosphorodithioate (not isolated)

Step 3

$$(C_2H_5O)_2P(S)SCH_2OH + C_2H_5SH \xrightarrow{50\ C} (C_2H_5O)_2P(S)SCH_2SC_2H_5 + H_2O$$

O, O-diethyl-S-(hyroxymethyl) phosphorodiethioate	ethyl mercaptan	O, O-diethyl-S-(ethylthiomethyl) phosphorodithioate PHORATE	water

FIGURE B-24. SYNTHESIS OF PHORATE

TABLE B- 40. REACTIONS OCCURRING DURING PRODUCTION OF PHORATE

Reaction Number	Generalized Reaction[a]
1	$4ROH + P_2S_5 \longrightarrow 2(RO)_2P(S)SH + H_2S$
2	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)SR' + H_2O$
3	$(RY)_2P(Y)SH + R'OH \longrightarrow (RY)_2P(Y)OR' + H_2S$
4	$(RY)_2P(Y)SR + R'OH \rightleftharpoons (RY)_2P(Y)OR' + RSH$
5	$(RY)_2P(Y)OR \xrightarrow{P_2S_5} (RY)_2P(Y)SR$
6	$(RY)_2P(Y)SH + H_2O \longrightarrow (RY)_2P(Y)OH + H_2S$
7	$(RY)_2P(S)OH \rightleftharpoons (RY)_2P(O)SH$
8	$2(RY)_2P(Y)YH \longrightarrow (RY)_2P(Y)YP(Y)(RY)_2 + H_2Y$
9	$2(RY)_2P(Y)SH \xrightarrow{[O]} (RY)_2P(Y)SSP(Y)(RY)_2$
10	$(RY)_2P(S)OR' \rightleftharpoons (RY)_2P(O)SR'$
11	$(RY)_2P(S)SH \longrightarrow (RY)_2PSH + S$
12	$(RY)_2PSH + R'OH \longrightarrow (RY)_2POR' + H_2S$
13	$(RY)_2PSH + R'OH \longrightarrow (RY)_2PSR' + H_2O$
14	$2(RY)_2PSH \xrightarrow{[O]} (RY)_2P(S)SP(YR)_2$
15	$(RY)_3PS \xrightarrow{[O]} (RY)_3PO$
16	$(RY)_2P(Y)SR + H_2O \longrightarrow (RY)_2P(Y)SH + ROH$
17	$(RY)_2P(Y)SR + H_2O \longrightarrow (RY)_2P(Y)OH + RSH$
18	$(RY)_2P(Y)OR + H_2O \longrightarrow (RY)_2P(Y)OH + ROH$
19	$(RY)_2P(Y)SH + CH_2O \longrightarrow (RY)_2P(Y)SCH_2OH$
20	$(RY)_2P(Y)SCH_2OH + RSH \longrightarrow (RY)_2P(Y)SCH_2SR + H_2O$
21	$(RY)_2P(Y)SCH_2OH + RSH \longrightarrow (RY)_2P(Y)SCH_2OR + H_2S$
22	$(RY)_2P(Y)SCH_2OH + ROH \longrightarrow (RY)_2P(Y)SCH_2OR + H_2O$
23	$RSH + CH_2O \rightleftharpoons RSCH_2OH$
24	$RSCH_2OH + RSH \longrightarrow RSCH_2SR + H_2O$
25	$RSCH_2OH + RSH \longrightarrow RSCH_2OR + H_2S$
26	$RSR \xrightarrow{[O]} RSOR$
27	$RSOR \xrightarrow{[O]} RSO_2R$
28	$2RSH \xrightarrow{[O]} RSSR$

a. R = alkyl or substituted alkyl; Y = O or S

TABLE B- 41. POSSIBLE CONTAMINANTS IN PHORATE

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	P_2S_5	SM	--	1	0
2	C_2H_5OH	SM	--	1	+
3	$(C_2H_5O)_2P(S)SH$	1+2 28	1 16	1 St	++
4	$(C_2H_5O)_2(C_2H_5S)PS$	3+2	2	1	++
5	$(C_2H_5O)_3PS$	3+2 4	3 4	1 1	++
6	$(C_2H_5O)(C_2H_5S)_2PS$	4 4	5 4	1 1	+
7	$(C_2H_5O)(C_2H_5S)P(S)SH$	3 3	5 4	1 1	+
8	$(C_2H_5O)_2P(S)OH$	3 28	6 17	1 St	+
9	$(C_2H_5O)_2P(O)SH$	8	7	1 or St	+
10	$(C_2H_5O)_2P(O)OH$	9	6	1 or St	+
11	$(C_2H_5O)(C_2H_5S)P(O)SH$	7	7	1	0
12	$(C_2H_5S)_2P(S)SH$	7 7	5 4	1 1	0
13	$(C_2H_5O)_2P(S)SP(S)(OC_2H_5)_2$	3	8	1	+
14	$(C_2H_5O)_2P(S)SSP(S)(OC_2H_5)_2$	3	9	1	0
15	$(C_2H_5O)_2PSH$	3	11	1	0
16	$(C_2H_5O)_3P$	15	12	1	0
17	$(C_2H_5O)_2(C_2H_5S)PO$	15	13	1	0

TABLE B-41. (Continued)

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
18	$(C_2H_5O)_2P(S)SP(OC_2H_5)_2$	15	14	1	0
19	$(C_2H_5O)_2(C_2H_5S)PO$	5	10	1	+
20	$(C_2H_5O)_3PO$	19 5	4 15	1	0
21	$(C_2H_5S)_3PS$	6	4	1	0
22	$(C_2H_5S)_3PO$	21	15	1	0
23	$(C_2H_5O)(C_2H_5S)_2PO$	6	15	1	0
24	C_2H_5SH	SM	--	3	0
25	$C_2H_5SSC_2H_5$	24	28	3	+
26	$(C_2H_5O)_2P(S)SCH_2OH$	3	19	2	++
27	$(C_2H_5O)(C_2H_5S)P(S)SCH_2OH$	7	19	2	+
28	$(C_2H_5O)_2P(S)SCH_2SC_2H_5$	26+24	20	3	PHORATE
29	$(C_2H_5O)(C_2H_5S)P(S)SCH_2SC_2H_5$	27+24	20	3	++
30	$(C_2H_5O)_2P(S)SCH_2OC_2H_5$	26+24	21	3	++
31	$(C_2H_5O)(C_2H_5S)P(S)SCH_2OC_2H_5$	27+24	21	3	+
32	$[(C_2H_5O)_2P(S)SCH_2]_2O$	26	22	2	+
33	$[(C_2H_5O)_2P(S)S]_2CH_2$	26+3	2	2	+
34	$(C_2H_5O)_2P(S)SCH_2OP(S)OC_2H_5)_2$	26+3	3	2	+
35	$C_2H_5SCH_2OH$	24	23	3	0

TABLE B-41. (Continued)

No.	Contaminant	Pre-cursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
36	$C_2H_5SCH_2SC_2H_5$	35+24	24	3	+
37	$C_2H_5SCH_2OC_2H_5$	35+24	25	3	+
38	$(C_2H_5O)_2P(S)SCH_2OCH_2SC_2H_5$	26+35	22	3	+
39	$(C_2H_5O)_2P(O)SCH_2SC_2H_5$	28	15	St	+
40	$(C_2H_5O)_2P(S)SCH_2SOC_2H_5$	28	26	St	+
41	$(C_2H_5O)_2P(S)SCH_2SO_2C_2H_5$	40	27	St	+
42	$(C_2H_5O)_2P(O)SCH_2SOC_2H_5$	39	26	St	0
43	$(C_2H_5O)_2P(O)SCH_2SO_2C_2H_5$	42	27	St	0
44	$C_2H_5SCH_2SH$	28	17	St	+
45	$(C_2H_5S)_2P(S)SCH_2OH$	27+24	4	3	0
46	$(C_2H_5S)_2P(S)SCH_2SC_2H_5$	45+24	20	3	+
47	$(C_2H_5S)_2P(S)SCH_2OC_2H_5$	45+24	21	3	0
48	$(C_2H_5O)_2P(S)OCH_2SC_2H_5$	3+35	3	3	+
49	$(C_2H_5O)_2PSCH_2SC_2H_5$	15+35	13	3	+

a. Precursor refers to the contaminant numbered in this table. SM = starting material.

b. See Table B-40.

c. The step (Figure B-24) during which the contaminant might be formed; St indicates possible formation during storage.

d. 0 = likely to be present at levels below 1 ppm.
+ = likely to be present at levels above 1 ppm but below 100 ppm.
++ = likely to be present at levels above 100 ppm.

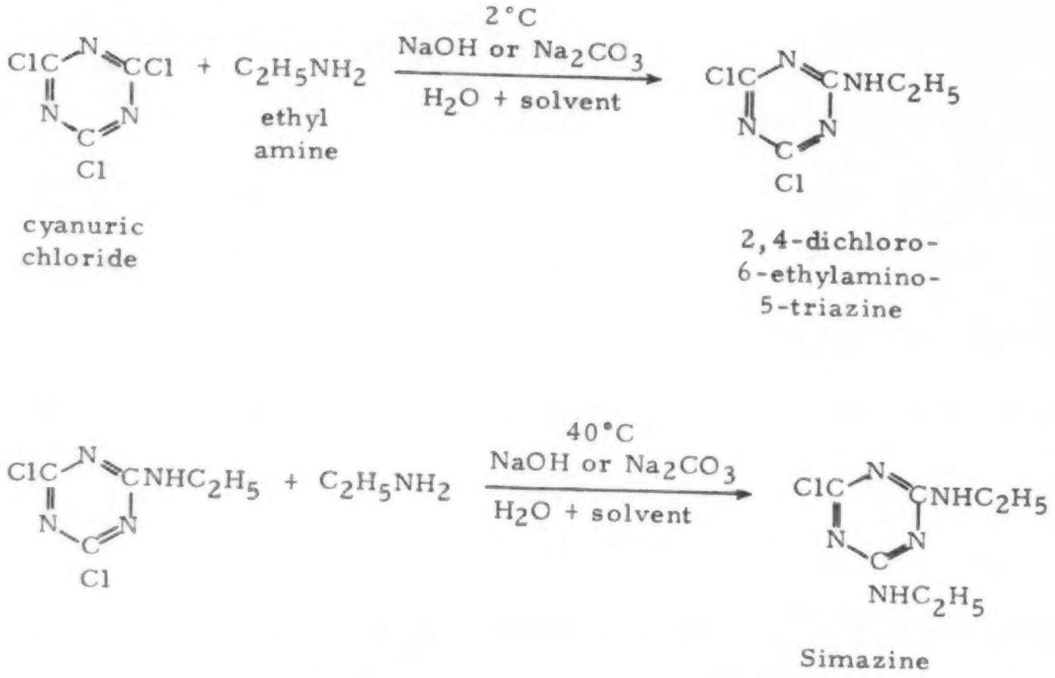

2°C
NaOH or Na2CO3
H2O + solvent
ClC
N
CCl
Cl
C2H5NH2
ethyl amine
cyanuric chloride
CNHC2H5
2,4-dichloro-6-ethylamino-5-triazine
40°C
NaOH or Na2CO3
H2O + solvent
NHC2H5
Simazine

FIGURE B-25. SYNTHESIS OF SIMAZINE

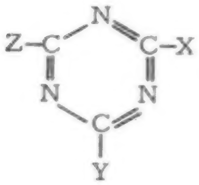
TABLE B-42. REACTIONS OCCURRING DURING SIMAZINE PRODUCTION

Reaction No.	Generalized Reaction[a]
1	$RCl + R'R''NH \xrightarrow{OH^-} RNR'R''$
2	$RCl + H_2O \xrightarrow{OH^-} ROH$
3	$>C = N- + H_2O \longrightarrow >C = O + -NH_2$
4	$>C = N- + R'R''NH \longrightarrow >C = NR' + -NHR''$
5	$RCl + RNHR' \xrightarrow{OH^-} R_2NR'$

(a) R = a substituted 1, 3, 5-triazine group; R' and R'' = C_2H_5 or H.

TABLE B-43. POSSIBLE CONTAMINANTS IN SIMAZINE

No.	Contaminant	Precursor(a)	Reaction Number(b)	Likelihood of Being Present in Final Product(c)
1	$C_2H_5NH_2$	Starting Material		+
2	$(C_2H_5)_2NH$	Impurity in 1		o
3	NH_3	Impurity in 1		o



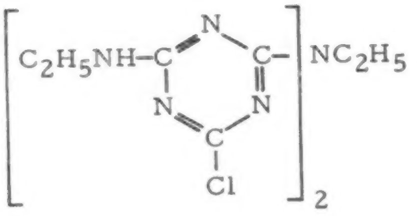
with the following substituents

No.	X=	Y=	Z=	Precursor(a)	Reaction Number(b)	Likelihood of Being Present in Final Product(c)
4	Cl	Cl	Cl	Starting Material		+
5	Cl	Cl	C_2H_5NH	4 + 1	1	++
6	Cl	C_2H_5NH	C_2H_5NH	5 + 1	1	Simazine
7	C_2H_5NH	C_2H_5NH	C_2H_5NH	6 + 1	1	++
8	Cl	Cl	$(C_2H_5)_2N$	4 + 2	1	o
9	Cl	$(C_2H_5)_2N$	$(C_2H_5)_2N$	8 + 2	1	o
10	$(C_2H_5)_2N$	$(C_2H_5)_2N$	$(C_2H_5)_2N$	9 + 2	1	o
11	Cl	Cl	NH_2	4 + 3	1	o
12	Cl	NH_2	NH_2	11 + 3	1	o
13	NH_2	NH_2	NH_2	12 + 3	1	o
14	Cl	$(C_2H_5)_2N$	C_2H_5NH	5 + 2	1	++
15	Cl	NH_2	C_2H_5NH	5 + 3	1	++

TABLE B-43. (Continued)

No.	Contaminant			Precursor[(a)]	Reaction Number[(b)]	Likelihood of Being Present in Final Product[(c)]
	X=	Y=	Z=			
16	$(C_2H_5)_2N$	C_2H_5NH	C_2H_5NH	6 + 2	1	o
17	NH_2	C_2H_5NH	C_2H_5NH	6 + 3	1	o
18	Cl	NH_2	$(C_2H_5)_2N$	8 + 3	1	o
19	NH_2	$(C_2H_5)_2N$	C_2H_5NH	14 + 3	1	o
20	$(C_2H_5)_2N$	$(C_2H_5)_2N$	C_2H_5NH	14 + 2	1	o
21	NH_2	$(C_2H_5)_2N$	$(C_2H_5)_2N$	9 + 3	1	o
22	C_2H_5NH	NH_2	NH_2	12 + 1	1	o
23	$(C_2H_5)_2H$	NH_2	NH_2	12 + 2	1	o
24	Cl	Cl	OH	4	2	+
25	Cl	OH	OH	24	2	+
26	OH	OH	OH	25	2	+
27	Cl	OH	C_2H_5NH	5	2	++
28	OH	OH	C_2H_5NH	27	2	++
29	OH	C_2H_5NH	C_2H_5NH	6	2	++
30	Cl	OH	$(C_2H_5)_2N$	8	2	o
31	OH	OH	$(C_2H_5)_2N$	31	2	o
32	OH	$(C_2H_5)_2N$	$(C_2H_5)_2N$	9	2	o
33	Cl	OH	NH_2	11	2	o
34	OH	OH	NH_2	33	2	o
35	OH	NH_2	NH_2	12	2	o

TABLE B-43. (Continued)

No.	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Likelihood of Being Present in Final Product[(c)]
36	OH $(C_2H_5)_2N$ C_2H_5NH	14	2	0
37	OH NH_2 C_2H_5NH	15	2	0
38	OH NH_2 $(C_2H_5)_2N$	18	2	0
39	$C_2H_5NHCON{=}C(NHC_2H_5)N{=}C(NHC_2H_5)NH_2$	6	3	0
40	$C_2H_5NHCON{=}C(NHC_2H_5)NH_2$	39	3	+
41	$C_2H_5NHCONH_2$	39	3	+
42	NH_2CONH_2	15	3	+
43	$(C_2H_5)_2NCONH_2$	14	3	+
44	$C_2H_5NHC(=NC_2H_5)NH_2$	6 + 1	4	+
45	$NH_2C(=NC_2H_5)NH_2$	15 + 1	4	0
46	$(C_2H_5)_2NC(=NC_2H_5)NH_2$	14 + 1	4	0
47		6 + 5	5	+

(a) Precursor refers to the contaminant numbered in this table.

(b) See Table B-42.

(c) 0 = likely present only at levels below 1 ppm
+ = likely present at levels above 1 ppm but below 100 ppm
++ = likely present at levels above 100 ppm.

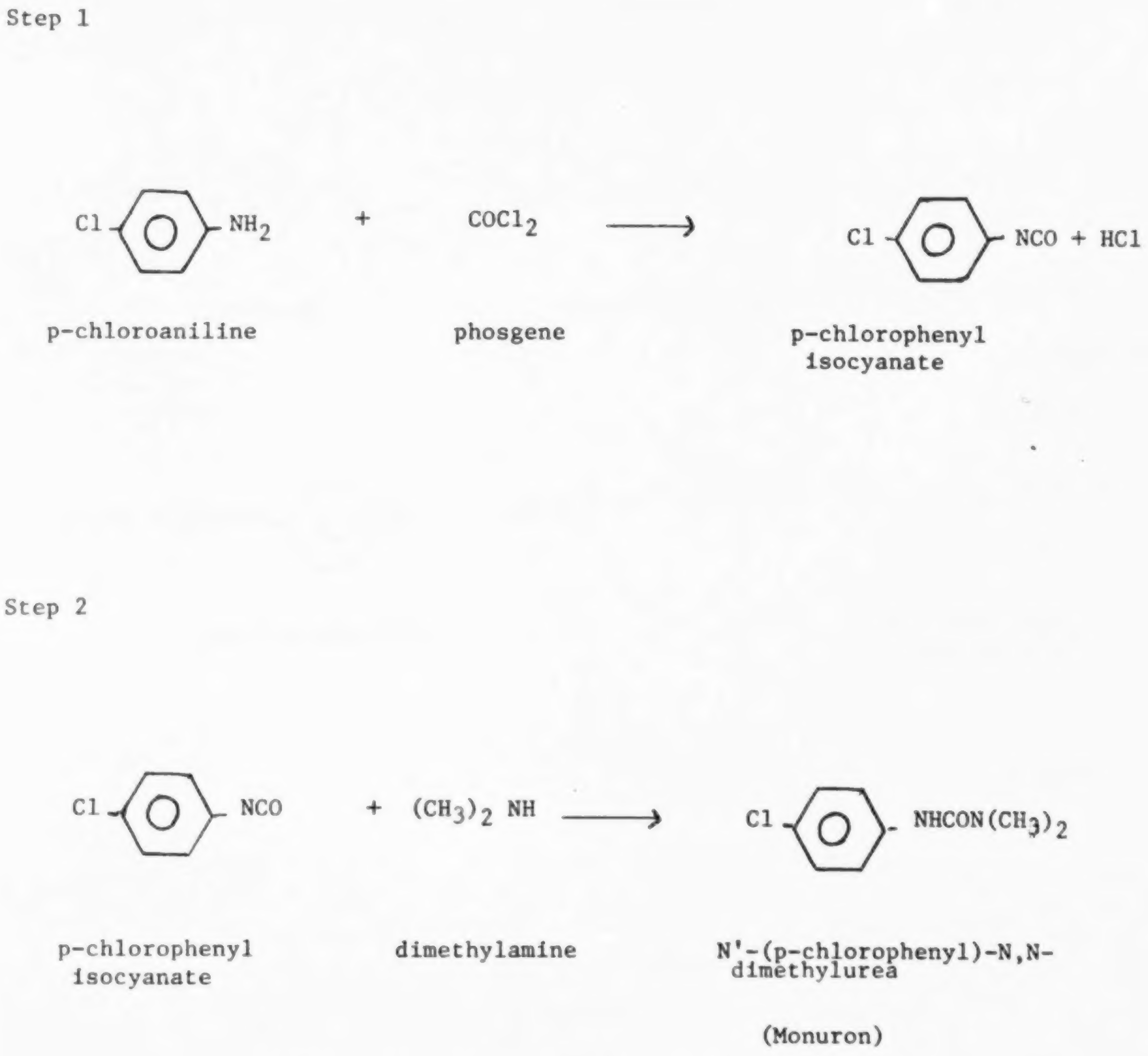

Step 1
Cl
NH2
+
COCl2
Cl
NCO + HCl
p-chloroaniline
phosgene
p-chlorophenyl isocyanate
Step 2
Cl
NCO
+ (CH3)2 NH
Cl
NHCON(CH3)2
p-chlorophenyl isocyanate
dimethylamine
N'-(p-chlorophenyl)-N,N-dimethylurea
(Monuron)

FIGURE B-26. SYNTHESIS OF MONURON

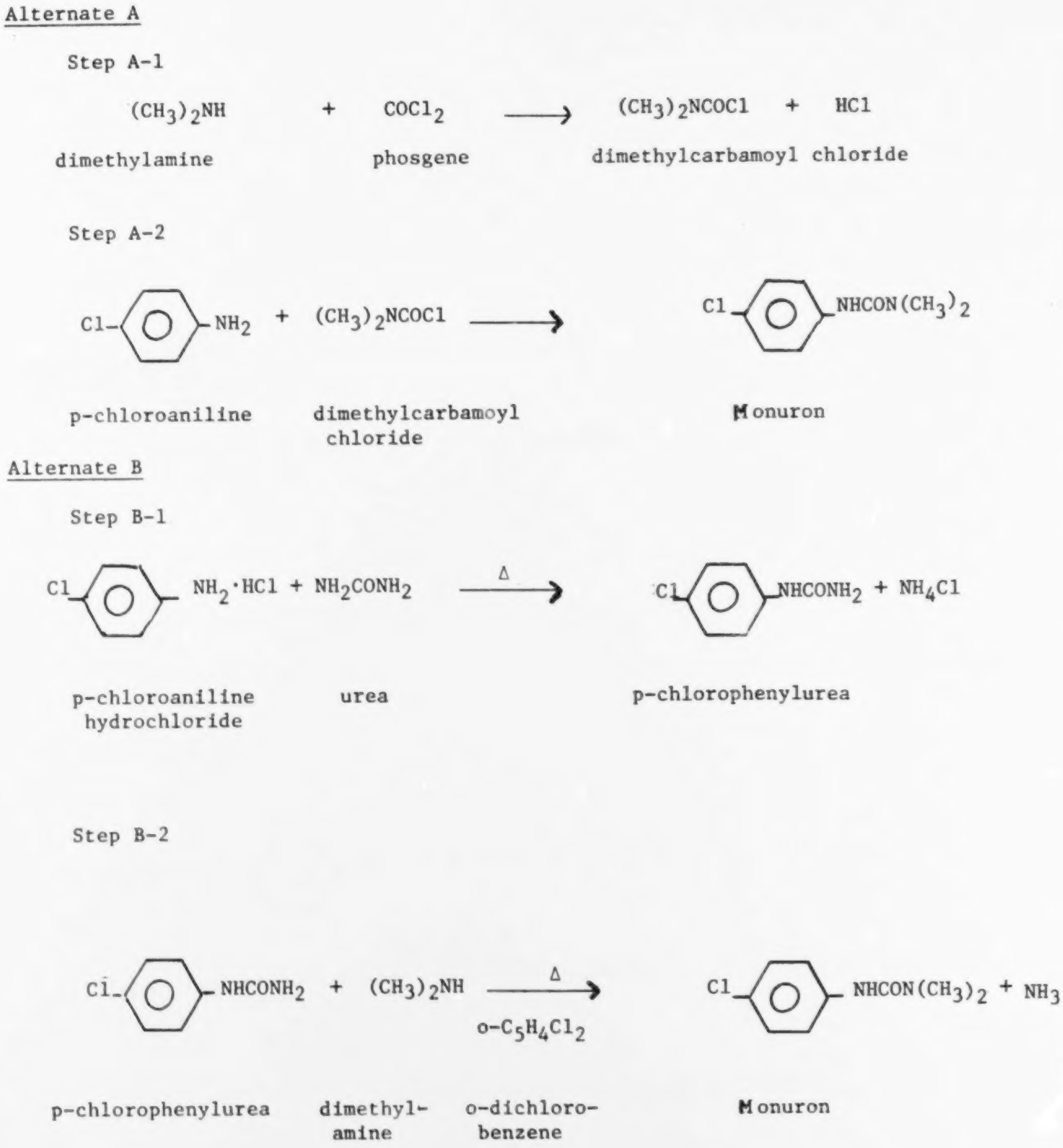
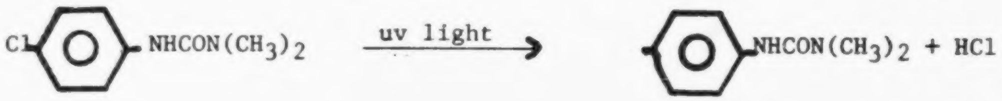
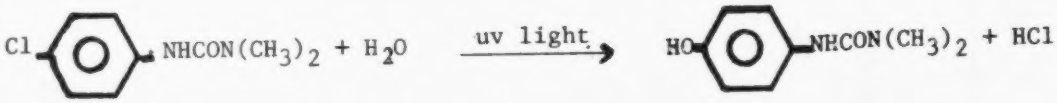
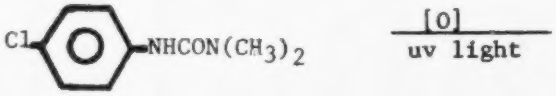

Alternate A
Step A-1
(CH3)2NH + COCl2 → (CH3)2NCOCl + HCl
dimethylamine
phosgene
dimethylcarbamoyl chloride
Step A-2
Cl
NH2
+ (CH3)2NCOCl
NHCON(CH3)2
p-chloroaniline
dimethylcarbamoyl chloride
Monuron
Alternate B
Step B-1
Cl
NH2·HCl + NH2CONH2
Δ
NHCONH2 + NH4Cl
p-chloroaniline hydrochloride
urea
p-chlorophenylurea
Step B-2
Cl
NHCONH2 + (CH3)2NH
Δ
o-C5H4Cl2
NHCON(CH3)2 + NH3
p-chlorophenylurea
dimethyl-amine
o-dichloro-benzene
Monuron

FIGURE B-27. ALTERNATE SYNTHESES OF MONURON

TABLE B-44. REACTIONS OCCURRING DURING PRODUCTION OF MONURON

Number	Generalized Reaction[(a)]
1	$\mathrm{RNH_2 + COCl_2 \longrightarrow RNHCOCl + HCL}$
2	$\mathrm{RNHCOCl \longrightarrow RNCO + HCl}$
3	$\mathrm{RNHCOCl + RNH_2 \longrightarrow RNHCONHR}$
4	$\mathrm{RNCO + R'_2NH \longrightarrow RNHCONR'_2}$
5	$\mathrm{RNCO + H_2O \longrightarrow RNH_2 + CO_2}$
6	$\mathrm{R'_2NH + COCl_2 \longrightarrow R'_2NCOCl + HCl}$
7	$\mathrm{RNH_2 + R'_2NCOCl \longrightarrow RNHCONR'_2 + HCl}$
8	$\mathrm{2R'_2NH + COCl_2 \longrightarrow R'_2NCONR'_2 + 2HCl}$
9	$\mathrm{RNH_2 \cdot HCl + NH_2CONH_2 \longrightarrow RNHCONH_2 + NH_4Cl}$
10	$\mathrm{RNHCONH_2 + R'_2NH \longrightarrow RNHCONR'_2 + NH_3}$
11	$\mathrm{RNHCONR'_2 + H_2O \longrightarrow RNH'_2 + R'_2NH + CO_2}$
12	 Cl NHCON(CH$_3$)$_2$ uv light NHCON(CH$_3$)$_2$ + HCl
13	 Cl NHCON(CH$_3$)$_2$ + H$_2$O uv light HO NHCON(CH$_3$)$_2$ + HCl
14	 **Photolysis products including:** $\mathrm{4\text{-}ClC_6H_4NHCON(CHO)CH_3}$ $\mathrm{4\text{-}ClC_6H_4NHCONHCHO}$ $\mathrm{4\text{-}ClC_6H_4NHCONHCH_3}$ $\mathrm{4\text{-}ClC_6H_4NHCONH_2}$ $\mathrm{4\text{-}ClC_6H_4NHCONHC_6H_4Cl\text{-}4}$ $\mathrm{4\text{-}ClC_6H_4NH_2}$ $\mathrm{4\text{-}ClC_6H_4NHCHO}$ $\mathrm{4\text{-}Cl\text{-}2\text{-}OH\text{-}C_6H_3NHCON(CH_3)_2}$

(a) R = aryl or methyl.
R′ = hydrogen or methyl

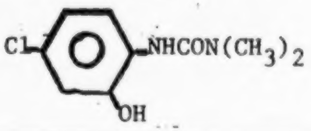
TABLE B-45. POSSIBLE CONTAMINANTS IN MONURON

Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
1	p-$ClC_6H_4NH_2$	SM 7 21	-- 5 11	1 2 St	++
2	$COCl_2$	SM	--	1	0
3	o-$ClC_6H_2NH_2$	Im-1	--	--	+
4	m-$ClC_6H_4NH_2$	Im-1	--	--	0
5	p-$ClC_6H_4NO_2$	Im-1	--	--	+
6	o-$ClC_6H_4NO_2$	Im-1	--	--	0
7	p-ClC_6H_4NCO	1+2	1+2	1	0
8	o-ClC_6H_4NCO	2+3	1+2	1	0
9	m-ClC_6H_4NCO	2+4	1+2	1	0
10	2,3-$Cl_2C_6H_3NH$	Im-1	--	--	0
11	2,5-$Cl_2C_6H_3NH$	Im-1	--	--	0
12	3,4-$Cl_2C_6H_3NH_2$	Im-1	--	--	0
13	2,4,5-$Cl_3C_6H_2NH_2$	Im-1	--	--	0
14	4-$ClC_6H_4NHCONHC_6H_4CL$-4	1+2 21	1+3 14	1 St	++

TABLE B-45. (Continued)

Number	Contaminant	Precursor[(a)]	Reaction Number[(b)]	Step[(c)]	Likelihood of Being Present in Final Product[(d)]
15	$2\text{-}ClC_6H_4NHCONHC_6H_4Cl\text{-}2$	2+3	1+3	1	0
16	$2\text{-}ClC_6H_4NHCONHC_6H_4Cl\text{-}4$	1+2+3	1+3	1	+
17	$3,4\text{-}Cl_2C_6H_3NHCONHC_6H_4Cl\text{-}4$	1+2+12	1+3	1	0
18	$(CH_3)_2NH$	SM 21	-- 11	2 St	+
19	CH_3NH_2	Im-18	--	--	0
20	NH_3	Im-18	--	--	0
21	$4\text{-}ClC_6H_4NHCON(CH_3)_2$	7+18 1+30 18+23	4 7 10	2 A-2 B-2	Monuron
22	$4\text{-}ClC_6H_4NHCONHCH_3$	7+19 21	4 14	2 St	++
23	$4\text{-}ClC_6H_4NHCONH_2$	7+20 1+33 21	4 9 14	2 B-1 St	+
24	$2\text{-}ClC_6H_4NHCON(CH_3)_2$	8+18	4	2	++
25	$2\text{-}ClC_6H_4NHCONHCH_3$	8+19	4	2	0
26	$2\text{-}ClC_6H_4NHCONH_2$	8+20 3+33	4 9	2 B-1	0

TABLE B-45 (Continued)

Number	Contaminant	Precursor[a]	Reaction Number[b]	Step[c]	Likelihood of Being Present in Final Product[d]
27	3-$ClC_6H_4NHCON(CH_3)_2$	9+18	4	2	0
28	$(C_2H_5)_3N$	Cat.	--	2	0
29	C_5H_5N	Cat.	--	2 or A-2	0
30	$(CH_3)_2NCOCl$	2+18	6	A-1	0
31	$(CH_3)_2NCON(CH_3)_2$	2+18	8	A-1	+
32	$(CH_3)_2NCONHCH_3$	19+30	7	A-1	0
33	NH_2CONH_2	SM	--	B-1	+
34	0-$C_6H_4Cl_2$	Sol.	--	B-2	+
35	$C_6H_5NHCON(CH_3)_2$	21	12	St	0
36	4-OH-$C_6H_4NHCON(CH_3)_2$	21	13	St	0
37	4-ClC_6H_4NH-$CON(CHO)CH_3$	21	14	St	0
38	4-$ClC_6H_4NHCONHCHO$	21	14	St	0
39	4-ClC_6H_4NHCHO	21	14	St	0
40		21	14	St	0

Footnotes appear on the following page.

Footnotes for Table B-45.

(a) Precursor refers to the contaminant numbered in this table
SM=starting material
Cat.= catalyst
Sol.= solvent
Im-1= impurity in No. 1
Im-18=impurity in No. 18

(b) See Table B- 44.

(c) The step (Fig. B-26 or Fig. B-27) during which the contaminant might be formed. St indicates possible formation during storage.

(d) o=likely to be present only at levels above 1 ppm
+=likely to be present only at levels above 1 ppm but below 100 ppm
++=likely to be present at levels above 100 ppm

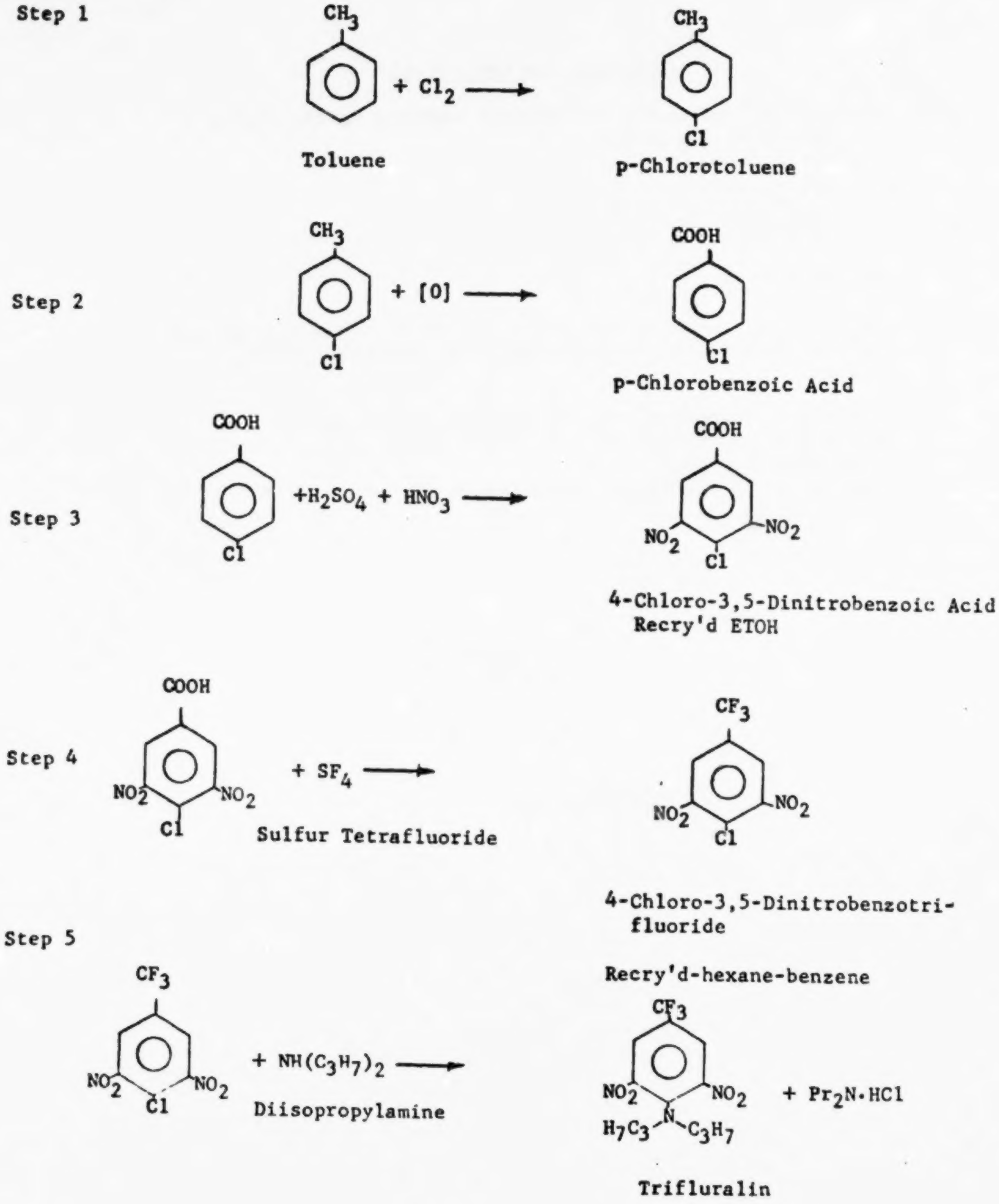

Step 1
CH_3
+ Cl_2
CH_3
Cl
Toluene
p-Chlorotoluene
Step 2
CH_3
+ [0]
COOH
Cl
Cl
p-Chlorobenzoic Acid
Step 3
COOH
$+H_2SO_4$ + HNO_3
COOH
NO_2
NO_2
Cl
Cl
4-Chloro-3,5-Dinitrobenzoic Acid
Recry'd ETOH
Step 4
COOH
NO_2
NO_2
Cl
+ SF_4
Sulfur Tetrafluoride
CF_3
NO_2
NO_2
Cl
4-Chloro-3,5-Dinitrobenzotrifluoride
Recry'd-hexane-benzene
Step 5
CF_3
NO_2
NO_2
Cl
+ $NH(C_3H_7)_2$
Diisopropylamine
CF_3
NO_2
NO_2
N
H_7C_3
C_3H_7
+ $Pr_2N \cdot HCl$
Trifluralin

FIGURE B-28. SYNTHESIS OF TRIFLURALIN

TABLE B-46. REACTIONS OCCURRING DURING PRODUCTION OF TRIFLURALIN

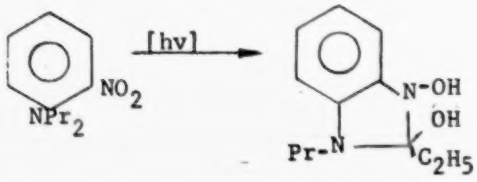
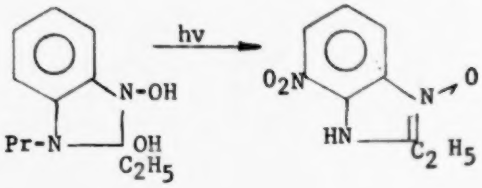
Reaction Number	Generalized Reaction
1	$ArH + Cl_2 \longrightarrow ArCl + HCl$
2	$ArCH_3 + Cl_2 \longrightarrow ArCH_2Cl + HCl$
3	$ArCH_3 \xrightarrow{[O]} ArCH_2OH$
4	$ArCH_2OH \xrightarrow{[O]} ArCHO$
5	$ArCHO \xrightarrow{[O]} ArCOOH$
6	$ArH \xrightarrow[H_2SO_4]{HNO_3} ArNO_2$
7	$ArCH_2OH \xrightarrow{SF_4} ArCH_2F$
8	$ArCHO \xrightarrow{SF_4} ArCHF_2$
9	$ArCOOH \xrightarrow{SF_4} ArCOF$
10	$ArCOOH \xrightarrow{SF_4} ArCF_3$
11	$ArCL + RR'NH \longrightarrow ArNRR'$
12	$ArCH_2Cl + RR'NH \longrightarrow ArCH_2NRR'$
13	$ArCOF + HNRR' \longrightarrow ArCONRR'$
14	$RR'NH + HX \longrightarrow RR'NH \cdot HX$
15	$ArCl + H_2O \xrightarrow{H^+} ArOH$
16	$ArCl + ROH \longrightarrow ArOR$
17	$ArNRR' \xrightarrow{[hv]} ArNHR$
18	$ArNHR \xrightarrow{[hv]} ArNH_2$
19	
20	 hv N-OH Pr-N OH C2H5 O2N N→O HN C2 H5

TABLE B-47. POSSIBLE CONTAMINANTS IN TRIFLURALIN

No	Contaminant(a)	Precursor(b)	Reaction(c) Number	Step(d)	Likelihood of Being Present in Final Product(e)
1	Toluene	SM	1	1	0
2	$C_6H_4(CH_3)Cl$	1	1	1	0
3	$C_6H_3(CH_3)Cl_2$	1	1	1	0
4	$C_6H_2(CH_3)Cl_3$	1	1	1	0
5	$C_6H_5CH_2Cl$	1	2	1	0
6	$C_6H_4(CH_2OH)Cl$	2	3	2	0
7	$C_6H_4(CHO)Cl$	2	4	2	0
8	$C_6H_4(COOH)Cl$	2	5	2	+
9	$C_6H_3(CH_2OH)Cl_2$	3	3	2	0
10	$C_6H_3(CHO)Cl_2$	3	4	2	0
11	$C_6H_3(COOH)Cl_2$	3	5	2	+
12	$C_6H_2(CH_2OH)Cl_3$	4	3	2	0
13	$C_6H_2(CHO)Cl_3$	4	4	2	0
14	$C_6H_2(COOH)Cl_3$	4	5	2	0
15	$C_6H_5CH_2OH$	5	3	2	0
16	C_6H_5CHO	5	4	2	0
17	C_6H_5COOH	5	5	2	0
18	$C_6H_3(NO_2)(COOH)Cl$	8	6	3	0
19	$C_6H_2(NO_2)_2(COOH)Cl$	8	6	3	+
20	$C_6H_3(NO_2)(CH_2OH)Cl$	6	6	3	0
21	$C_6H_2(NO_2)_2(CH_2OH)Cl$	6	6	3	0
22	$C_6H_3(NO_2)(CHO)Cl$	7	6	3	0
23	$C_6H_2(NO_2)_2(CHO)Cl$	7	6	3	0
24	$C_6H_2(NO_2)(COOH)Cl_2$	11	6	3	0

TABLE B-47. (Continued)

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
25	$C_6H(NO_2)_2(COOH)Cl_2$	11	6	3	+
26	$C_6H_2(NO_2)(CH_2OH)Cl_2$	9	6	3	0
27	$C_6H(NO_2)_2(CH_2OH)Cl_2$	9	6	3	0
28	$C_6H_2(NO_2)(CHO)Cl_2$	10	6	3	0
29	$C_6H(NO_2)_2(CHO)Cl_2$	10	6	3	0
30	$C_6H(NO_2)(COOH)Cl_3$	14	6	3	0
31	$C_6(NO_2)_2(COOH)Cl_3$	14	6	3	0
32	$C_6H(NO_2)(CH_2OH)Cl_3$	12	6	3	0
33	$C_6(NO_2)_2(CH_2OH)Cl_3$	12	6	3	0
34	$C_6H(NO_2)(CHO)Cl_3$	13	6	3	0
35	$C_6(NO_2)_2(CHO)Cl_3$	13	6	3	0
36	$C_6H_4(NO_2)CH_2OH$	15	6	3	0
37	$C_6H_3(NO_2)_2CH_2OH$	15	6	3	0
38	$C_6H_2(NO_2)_3CH_2OH$	15	6	3	0
39	$C_6H_4(NO_2)CHO$	16	6	3	0
40	$C_6H_3(NO_2)_2CHO$	16	6	3	0
41	$C_6H_4(NO_2)COOH$	17	6	3	0
42	$C_6H_3(NO_2)_2COOH$	17	6	3	0
43	$C_6H_3(NO_2)(COF)Cl$	18	9	4	0
44	$C_6H_3(NO_2)(CF_3)Cl$	18	10	4	+
45	$C_6H_2(NO_2)_2(COF)Cl$	19	9	4	0
46	$C_6H_2(NO_2)_2(CF_3)Cl$	19	10	4	++
47	$C_6H_3(NO_2)(CH_2F)Cl$	20	7	4	0

TABLE B-47. (Continued)

No.	Contaminant(a)	Precursor(b)	Reaction(c) Number	Step(d)	Likelihood of Being Present in Final Product(e)
48	$C_6H_2(NO_2)_2(CH_2F)Cl$	21	7	4	0
49	$C_6H_3(NO_2)(CHF_2)Cl$	22	8	4	0
50	$C_6H_2(NO_2)_2(CHF_2)Cl$	23	8	4	0
51	$C_6H_2(NO_2)(COF)Cl_2$	24	9	4	0
52	$C_6H_2(NO_2)(CF_3)Cl_2$	24	10	4	++
53	$C_6H(NO_2)_2(COF)Cl_2$	25	9	4	0
54	$C_6H(NO_2)_2(CF_3)Cl_2$	25	10	4	+
55	$C_6H_2(NO_2)(CH_2F)Cl_2$	26	7	4	0
56	$C_6H(NO_2)_2(CH_2F)Cl_2$	27	7	4	0
57	$C_6H_2(NO_2)(CHF_2)Cl_2$	28	8	4	0
58	$C_6H(NO_2)_2(CHF_2)Cl_2$	29	8	4	0
59	$C_6H(NO_2)(COF)Cl_3$	30	9	4	0
60	$C_6H(NO_2)(CF_3)Cl_3$	30	10	4	0
61	$C_6H(NO_2)_2(COF)Cl_3$	31	9	4	0
62	$C_6H(NO_2)_2(CF_3)Cl_3$	31	10	4	0
63	$C_6H(NO_2)(CH_2F)Cl_3$	32	7	4	0
64	$C_6(NO_2)_2(CH_2F)Cl_3$	33	7	4	0
65	$C_6H(NO_2)(CHF_2)Cl_3$	34	8	4	0
66	$C_6H(NO_2)(CHF_2)Cl_3$	35	8	4	0
67	$C_6H_4(NO_2)CH_2F$	36	7	4	0
68	$C_6H_3(NO_2)_2CH_2F$	37	7	4	0
69	$C_6H_2(NO_2)_3CH_2F$	38	7	4	0
70	$C_6H_4(NO_2)CHF_2$	39	8	4	0

TABLE B-47. (Continued)

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
71	$C_6H_3(NO_2)_2CHF_2$	40	8	4	0
72	$C_6H_4(NO_2)COF$	41	9	4	0
73	$C_6H_4(NO_2)CF_3$	41	10	4	0
74	$C_6H_3(NO_2)_2COF$	42	9	4	0
75	$C_6H_3(NO_2)_2CF_3$	42	10	4	0
76	Pa_2NH	SM	-	5	0
77	$Pr_2NH \cdot HCl$	76	14	5	+
78	$C_6H_3(NO_2)(CONPr_2)Cl$	43	13	5	0
79	$C_6H_3(NO_2)(COF)NPr_2$	43	11	5	0
80	$C_6H_3(NO_2)(CONPr_2)NPr_2$	43	11&13	5	0
81	$C_6H_3(NO_2)(CF_3)NPr_2$	44	11	5	++
82	$C_6H_2(NO_2)_2(CONPr_2)Cl$	45	13	5	0
83	$C_6H_2(NO_2)_2(COOH)NPr_2$	19	11	5	+
84	$C_6H_2(NO_2)_2(CONPr_2)NPr_2$	45	11&13	5	+
85	$C_6H_2(NO_2)_2(CF_3)NPr_2$	46	11	5	Trifluralin
86	$C_6H_3(NO_2)(CH_2F)NPr_2$	47	11	5	0
87	$C_6H_2(NO_2)_2(CH_2F)NPr_2$	48	11	5	0
88	$C_6H_3(NO_2)(CHF_2)NPr_2$	49	11	5	0
89	$C_6H_2(NO_2)_2(CHF_2)NPr_2$	50	11	5	0
90	$C_6H_2(NO_2)(CONPr_2)Cl_2$	51	13	5	0
91	$C_6H_2(NO_2)(COF)(NPr_2)Cl$	51	11	5	0
92	$C_6H_2(NO_2)(COF)(NPr_2)_2$	51	11	5	0
93	$C_6H_2(NO_2)(CONPr_2)(NPr_2)_2$	51	11&13	5	0
94	$C_6H_4(NO_2)(CF_3)(NPr_2)Cl$	52	11	5	+

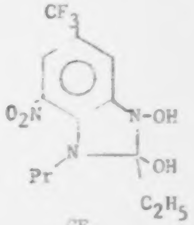
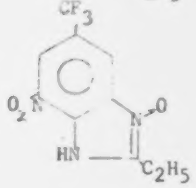
TABLE B-47. (Continued)

No.	Contaminant(a)	Precursor(b)	Reaction(c) Number	Step(d)	Likelihood of Being Present in Final Product(e)
95	$C_6H_4(NO_2)(CF_3)(NPr_2)_2$	52	11	5	+
96	$C_6H(NO_2)_2(CONPr_2)Cl_2$	53	13	5	0
97	$C_6H(NO_2)_2(CONPr_2)(NPr_2)Cl$	53	11&13	5	0
98	$C_6H(NO_2)_2(CONPr_2)(NPr_2)_2$	53	11&13	5	0
99	$C_6H(NO_2)_2(COF)(NPr_2)Cl$	53	11	5	0
100	$C_6H(NO_2)_2(CF_3)(NPr_2)Cl$	54	11	5	0
101	$C_6H(NO_2)_2(CF_3)(NPr_2)_2$	54	11	5	+
102	$C_6H_2(NO_2)(CH_2F)(NPr_2)_2Cl$	55	11	5	0
103	$C_6H_2(NO_2)(CH_2F)(NPr_2)_2$	55	11	5	0
104	$C_6H(NO_2)_2(CH_2F)(NPr_2)Cl$	56	11	5	0
105	$C_6H(NO_2)_2(CH_2F)(NPr_2)_2$	56	11	5	0
106	$C_6H_2(NO_2)(CHF_2)(NPr_2)Cl$	57	11	5	0
107	$C_6H_2(NO_2)(CHF_2)(NPr_2)_2$	57	11	5	0
108	$C_6H(NO_2)_2(CHF_2)(NPr_2)Cl$	58	11	5	0
109	$C_6H(NO_2)_2(CHF_2)(NPr_2)_2$	58	11	5	0
110	$C_6H(NO_2)(CONPr_2)Cl_3$	59	13	5	0
111	$C_6H(NO_2)(COF)(NPr_2)Cl_2$	59	11	5	0
112	$C_6H_4(NO_2)(COF)(NPr_2)_2Cl$	59	11	5	0
113	$C_6H_4(NO_2)(COF)(NPr_2)_3$	59	11	5	0
114	$C_6H_4(NO_2)(CONPr_2)(NPr_2)_3$	59	11&13	5	0
115	$C_6H(NO_2)(CF_3)(NPr_2)Cl_2$	60	11	5	0
116	$C_6H_4(NO_2)(CF_3)(NPr_2)_2Cl$	60	11	5	0

TABLE B-47. (Continued)

No.	Contaminant[(a)]	Precursor[(b)]	Reaction Number[(c)]	Step[(d)]	Likelihood of Being Present in Final Product[(e)]
117	$C_6H_4(NO_2)(CF_3)(NPr_2)_3$	60	11	5	0
118	$C_6H(NO_2)_2(CONPr_2)Cl_3$	61	13	5	0
119	$C_6H(NO_2)_2(COF)(NPr_2)Cl_2$	61	11	5	0
120	$C_6H(NO_2)_2(COF)(NPr_2)_2Cl$	61	11	5	0
121	$C_6H(NO_2)_2(COF)(NPr_2)_3$	61	11	5	0
122	$C_6H(NO_2)_2(CONPr_2)(NPr_2)_3$	61	11&13	5	0
123	$C_6H(NO_2)_2(CF_3)(NPr_2)Cl_2$	62	11	5	0
124	$C_6H(NO_2)(CF_3)(NPr_2)_2Cl$	62	11	5	0
125	$C_6H_4(NO_2)(CF_3)(NPr_2)_3$	62	11	5	0
126	$C_6H(NO_2)(CH_2F)(NPr_2)Cl_2$	63	11	5	0
127	$C_6H(NO_2)(CH_2F)(NPr_2)_2Cl$	63	11	5	0
128	$C_6H(NO_2)(CH_2F)(NPr_2)_3$	63	11	5	0
129	$C_6(NO_2)_2(CH_2F)(NPr_2)Cl_2$	64	11	5	0
130	$C_6(NO_2)_2(CH_2F)(NPr_2)_2Cl$	64	11	5	0
131	$C_6(NO_2)_2(CH_2F)(NPr_2)_3$	64	11	5	0
132	$C_6H(NO_2)(CHF_2)(NPr_2)Cl_2$	65	11	5	0
133	$C_6H(NO_2)(CHF_2)(NPr_2)_2Cl$	65	11	5	0
134	$C_6H(NO_2)(CHF_2)(NPr_2)_3$	65	11	5	0
135	$C_6(NO_2)_2(CHF_2)(NPr_2)Cl_2$	66	11	5	0
136	$C_6(NO_2)_2(CHF_2)(NPr_2)_2Cl$	66	11	5	0
137	$C_6(NO_2)_2(CHF_2)(NPr_2)_3$	66	11	5	0
138	$C_6H_4(NO_2)(CONPr_2)$	72	13	5	0

TABLE B-47. (Continued)

No.	Contaminant[a]	Precursor[b]	Reaction Number[c]	Step[d]	Likelihood of Being Present in Final Product[e]
139	$C_6H_3(NO_2)_2(CONPr_2)$	74	13	5	0
140	$C_6H_2(NO_2)_2(CF_3)OH$	46	15	3,5	++
141	$C_6H_2(NO_2)_2(CF_3)OC_2H_5$	46	16	3	+
142	$C_6H_5CH_2NPr_2$	5	12	5	0
143	$C_6H_2(NO_2)_2(CF_3)NHPr$	85	17	5	+
144	$C_6H_2(NO_2)_2(CF_3)NH_2$	143	18	5	+
145		85	19	5	+
146		145	20	5	+

(a) There are a number of different positional isomers possible for many of the contaminants.

(b) Precursor refers to the contaminant numbered in this table SM=starting material.

(c) See Table B-46.

(d) The step (Figure B-28) during which the contaminant might be formed.

(e) 0 = likely to be present below 1 ppm.
+ = likely to be present above 1 ppm but below 100 ppm.
++ = likely to be present at levels above 100 ppm.